Experimental Investigations and Simulation of Field Aging of Bituminous Concrete for Indian Conditions Using Modified Oven

THESIS

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CERTIFICATE

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DECLARATION

This is to certify that the thesis titled "**Experimental Investigations and Simulation of Field Aging of Bituminous Concrete for Indian Conditions Using Modified Oven"** is based on my own research work and has been carried out under the guidance and supervision of Prof. **V. VINAYAKA RAM**, Associate Professor, Dept. of Civil Engineering, BITS Pilani, Hyderabad Campus, India and co-supervision of Prof. **SRIDHAR RAJU**, Associate Professor, Dept. of Civil Engineering, BITS Pilani, Hyderabad Campus, Hyderabad, India.

The data and information which I have used from various sources have been duly acknowledged. I declare that this work has not been previously submitted by me to any other university / institute for the award of any other degree or diploma.

Date: **Babu Sarath Kambham**

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ABSTRACT

Flexible pavement durability gets severely affected with short term as well as long term aging of bitumen in bituminous concrete mixtures. Laboratory simulation of the aging phenomenon always remained challenging. Many procedures have been developed during the last few decades to simulate the long term aging of bituminous concrete mixtures, of which AASHTO R30 has been the most popular laboratory based simulation procedure of long term aging of bituminous concrete mixtures. AASHTO R30 procedure involves a conventional oven, capable of maintaining temperatures up to 200 ° C for required period of 5 days.

In the current research study, realizing the need to modify the oven for more efficient simulation than what was possible with conventional oven, a modified oven was developed with an additional capability of supplying oxygen for an extended period of exposure. This modified oven was employed to artificially age the bituminous concrete specimens, prepared in the laboratory with the same mixture specification as those collected from the field. In addition, a few more samples with warm mix additive (Sasobit) and filler (hydrated lime) were also prepared and aged in the oven for checking their efficacy in delaying the aging phenomenon. These laboratory samples were subjected to aging from one to fifteen days (not limiting to the 5 days aging as recommended by AASHTO R30 guidelines) in the modified oven after initial short-term aging of the un-compacted bituminous concrete mixtures. The aged bituminous concrete samples were then tested to find Marshall's stability, flow value and tensile strength ratio (TSR).

Bitumen was later extracted from these artificially aged samples using Sohxlet bitumen extractor. Similarly, differently aged field cores were collected from two of the National highways (NH 16 and NH 65) in India. The bitumen was extracted, using Sohxlet extractor, from these field cores also for further testing.

The physical and microstructural analysis was performed on the extracted bitumen from the lab aged as well as field extracted cores. Softening point, ductility, penetration tests

were performed to investigate the changes in these basic properties with aging. In addition, rheological properties through Dynamic Shear Rheometer (DSR), viscosities through Brookfield's Rotational Viscometer, Fourier Transform Infrared Spectroscopy (FT-IR), Field Emission Scanning Electron Microscope (FE SEM), Energy Dispersive X-Ray analysis (EDX), and changes in asphaltene and maltene contents in the aged bitumen samples were carried out on the residual aged bitumen samples for assessing the microstructural changes and to get a clue about the aging phenomenon.

The results obtained were analyzed to validate the aging procedure used in the laboratory to simulate the field aging. The laboratory samples without the additives aged much faster than those prepared with optimized quantities of warm mix additive (Sasobit) and hydrated lime, when exposed to modified oven aging process, devised as per AASHTO R30 guidelines. From the comparison of field samples and the laboratory aged samples, it was found that the 5-day exposure (as suggested by AASHTO R30 guidelines) was not enough to simulate the field aging process. Looking at the progressive deterioration of the laboratory aged samples with increased exposures times, it was felt necessary to expose the samples for more period than that suggested in AASHTO R30 guidelines.

The correlation between the laboratory aging through modified oven and the actual field aging was established through fundamental and rheological properties along with microstructural investigations on extracted bitumen from both laboratory and field specimens. Aging indices, developed from the fundamental properties of extracted binder, were employed to assess the level of aging occurred in the bitumen samples. Sulfoxide and carbonyl indices, developed from the Fourier Transform Infrared (FT-IR) spectrums, plotted for the laboratory and field aged bitumen samples, were employed to assess the extent of aging occurred in the samples. Sulfoxide index was found to be more efficient in indicating the aging when compared with carbonyl index during the current research. Maltene to asphaltene ratio, computed from the results of SARA analysis was also found to be an efficient indicator for assessing the level of aging of extracted bitumen samples.

Another major observation was that, the binder extracted from the top 12 mm section of the field extracted specimen underwent major chemical and microstructural

transformation when compared with binder extracted from the bottom section of the field core, clearly establishing the fact that the top section ages more aggressively.

Key Words: Bitumen Field Aging, Modified Oven, AASHTO R30, Sasobit, Hydrated Lime, Bitumen Rheology, Carbonyl Index, Sulfoxide Index, SARA Analysis, Microstructural Investigations on Bitumen.

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LIST OF ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
AFM	Atomic Force Microscopy
ASTM	American Society for Testing and Materials
DSR	Dynamic Shear Rheometer
EDX	Energy Dispersive X-Ray
EN	Europeen de Normalisation
FE SEM	Field Emission Scanning Electron Microscope
FT - IR	Fourier Transform Infrared Spectroscopy
ITS	Indirect Tensile Strength
LTOA	Long Term Oven Aging
MoRT&H	Ministry of Road Transport and Highways
MRTFOT	Modified Rolling Thin Film Oven Test
MSCR	Multiple Stress Creep and Recovery
NH	National highway
PAV	Pressure Aging Vessel
PI	Penetration Index
PRP	Penetration Retention Percentage
RAP	Reclaimed Asphalt Pavements
RCAT	Rotating Cylinder Aging Test
RFT	Rotating Flask Test
RRT	Rapid Recovery Test

RTFOT	Rolling Thin Film Oven Test
SARA	Saturates, Aromatics, Resins and Asphaltene
SHRP	Strategic Highway Research Program
SPI	Softening Point Increment
STOA	Short Term Oven Aging
TFOT	Thin Film Oven Test
TSR	Tensile Strength Ratio Test
UV	Ultraviolet
VG	Viscosity Grade
WMA	Warm Mix Asphalt

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Bitumen, also known as asphalt, is usually a dark brown to black, hydrocarbon viscoelastic binding material, produced from crude petroleum oil, as a distillation residue. This distillation, when happens naturally, results in deposits like lake asphalt. Highways and roads alone consume 85% of total bitumen produced in the world (Asphalt Institute, 2002). Bitumen is fully soluble in toluene and is used as adhesive, waterproofing material and as a binder in flexible pavement construction. The bituminous concrete, a designed mixture, contains mineral aggregates, fillers and binder; the bitumen functions as a binder. Bitumen has a share of 4 % to 8 % by weight of the bituminous concrete mixture, while influencing the cost by about 25 % to 30 %, depending upon the type and quality of the mixture.

Aging of bitumen, happening through its exposure to climatic variations as well as traffic loads, is an important phenomenon that needs to be quantified, to understand the process of pavement deterioration in general (Baek et al. 2012). Also, it is a well-established fact that the durability of the bituminous pavements is majorly affected by age hardening, moisture and thermal related damages.

The aging of the bitumen can be described as a set of complex physio–chemical processes, which occur throughout the service life of the pavement (Durrieu et al. 2008). Short-term aging can happen in the bituminous concrete, during the processes of mixing and placing, due to volatilization of bitumen. However, the long-term aging happens due to steric hardening and oxidation during service life of the pavement in the field.

At high temperatures, it has been established that the elastic stiffness of the bituminous mixture reduces to such an extent that the top bituminous layer become weaker than the bottom granular layers with progressing time and exposure levels. Hagos (2008) has observed that majority of the pavement failures happen due to binder aging alone. Apart from temperature, the air void content of the bituminous concrete mix was found to be the next major factor causing the aging of bitumen. Generally, it can be understood that more the air voids, greater will be the oxidation related aging (Morian et al. 2013). It can be generally observed that the aging of bitumen causes reduced penetration values, increased softening point, enhanced viscosity and brittleness. Because of changes in chemistry of bitumen, progressive aging may be expected in the form of loss of elastic responses thus leading to premature failure of the bituminous concrete and reduced service life of the pavement.

Over the years, to counter the aging phenomenon of bitumen, many anti-aging techniques were developed with the main intention of reducing the ill effects of aging and enhancing the service life of the pavement. M/s Shell bitumen company used Styrene-Butadiene-Styrene (SBS), as a bitumen modifier to reduce the aging effect (Feng et al. 2012). Also, Iwanski et al. (2013) have observed that hydrated lime, an antioxidant, decreased the rate of aging of bitumen, thus contributing towards improved durability of the pavements. Pan et al (2014) found that graphite, as a bitumen admixture, has the capability of enhancing the anti-aging characteristics of bitumen. Zhang et al. (2010) have used montmorillonite as a bitumen admixture for improving the physical and rheological properties along with storage stability of bitumen. Another notable bitumen admixture tried by the researchers, to enhance the anti-aging performance of the binders, was crumb rubber (Ali et al. 2013).

Usually, bitumen aging is simulated in the laboratory using Thin Film Oven Test (TFOT) and Rolling Thin Film Oven Test (RTFOT) for simulating short term aging followed by Pressure Aging Vessel (PAV) for simulating long term aging. However, these tests are developed to age the bitumen in isolation and hence they may not really simulate the field aging of bitumen because the bitumen is present as an integral part of bituminous concrete and not in isolation. It was in this direction that many aging protocols for bituminous concrete have been proposed by the researchers across the globe, among which, AASHTO R30 has been found to be very popular. This aging protocol, in general has not been considered as a versatile protocol to adopt changing environmental variations.

Changes happening in chemistry, microstructure, physical properties and rheology in aged bituminous binders, are expected to provide critical clues about the dynamics of bitumen aging phenomenon. A wholesome approach of investigating the field aged and artificially aged bitumen samples is expected to result in more clarity in understating the aging phenomenon of bitumen, scientifically.

Multiple studies (Kim et al. 2015, Farrar et al. 2013) have been reported that the top 10 to 12 mm layer will have the maximum aging happening while the bottom section is minimally aged. An in-depth study on bitumen residue samples extracted from top and bottom slices and comparing them with the artificially aged samples would provide critical clues to understand this phenomenon more clearly. The process of short term aging of the loose mixture followed by the long term aging exposure of the compacted short term aged specimens is expected to replicate the field process more closely.

In the current era of preferential road building activity with recycled asphalt materials, understanding the kinetics of aging at different depths of the surface layer of the flexible pavement, becomes very useful. Also, the efficacy of warm mix additives in controlling the aging phenomena is expected to add value to the process of recycling. The reduced temperature at which the mixture acquires the working viscosity, in the presence of warm mix additives, is expected to have huge impact on the aging phenomenon due to the reason that the mixture undergoes relatively lower levels of short term aging effects.

1.2 Scope of study

The scope of present study is limited to collecting field cores from two different locations with similar environmental conditions and different ages of exposure. Also, it was ensured that the selected locations had the same grade of bituminous concrete mixtures being used as surface course. In other words, the study is limited to only one grade of Bituminous Concrete mixture. Also, the grade of the bitumen used at both these sites is straight run VG 30 bitumen without any modifications. This study is limited to study the efficacy of one popular warm mix additive Sasobit and hydrated lime as a filler and anti-stripping agent. The oven, modified to fulfill the requirements of AASHTO R30 guidelines was improvised with additional oxygen supply through cylinders and also with additional capability of raising the temperatures to suit the planned aging regimes.

1.3 Thesis organization

This thesis report consists of six chapters. The first chapter introduces the concept of bitumen aging and its influence on flexible pavement durability. The need for the present study, objectives and the scope of the work are presented in this chapter.

The second chapter consists of the summary of the comprehensive review being carried out on bitumen aging mechanism, factors affecting aging, different methods used for simulating aging in the lab and the measures that are to be taken to reduce the aging phenomenon. Microstructural studies that can be done on bitumen are also discussed thoroughly. The research gaps are identified and research objectives are presented at the end of this chapter.

Chapter three deals with research methodology that is followed in the present study. Each stage of research is explained in detail.

The fourth chapter consists of the experimental methods and materials that are used for this research. This chapter explains different aging techniques being used during the current research. The methods used for laboratory specimen preparation and material procurement are presented. The development of oven aging technique is discussed. The extraction of bitumen from the lab aged and field specimens is also presented. Details regarding all the micro structural and rheological studies were also presented in this chapter

In the fifth chapter, the results of tests conducted on bitumen and bituminous concrete before and after aging are presented. The results of microstructural investigations that are carried out on the samples are also discussed. The interpretation and summary of results is discussed at the end.

The last chapter i.e. sixth chapter contains the conclusions, specific contributions and the scope for further research. This chapter is followed with the references and list of publications. The biography of the scholar, supervisor and co-supervisor are presented at the end.

CHAPTER 2

BITUMEN AND BITUMINOUS CONCRETE AGING – A REVIEW

2.1 Introduction

A detailed literature review was carried out in the main domain of aging of surface layer of flexible pavements with emphasis on topics namely, factors affecting bitumen aging. Methods of controlling aging, methods for simulation of long term aging in laboratory and microstructural investigations carried out on aged bituminous concrete samples are discussed.

2.2 Bitumen aging

The failures in pavements are caused by many load and environmental related factors. Under the environmental related factors, two major factors are moisture damage and the age hardening. In this current research, the age hardening or bitumen aging aspect is being addressed.

It is a well-known fact that the initial aging up to certain level is beneficial as it helps the pavement in load spreading ability and improved resistance to deformation of the pavement structure. This phenomenon is called curing and it helps in increasing the life of pavement. However, further aging will result in loss of elastic responses, causing premature failure of the pavement structure in general and the surface course in particular. Aggressive environmental conditions have always affected the durability of the bituminous concrete pavements and hence, understanding the field aging phenomenon of bitumen has become very important. The aging in the bitumen pavements can be divided into the short term and long term aging phenomenon.

- **Short Term Aging:** This type of aging occurs during mixing and laying of bituminous concrete. During mixing of bituminous concrete, to ensure a workable viscosity, the temperature of an unmodified bitumen is maintained at 150° C while the aggregates are heated up to 180° C. Dominant presence of aggregates at higher temperatures will result in active heat transfer happening from aggregate to bitumen, raising the temperature of bitumen further. This results in bitumen losing its volatile compounds which in turn, results in oxidation process.

- **Long Term Aging:** This type of aging occurs during the service life of the flexible pavement. The service life of top layers of a flexible pavement is usually around 5 – 7 years. During this period, the oxidation and the loss of volatile substances will occur, causing the aging of bitumen (Robert et al. 2015). The rate of aging in long term aging is much lesser when compared with short term aging, as depicted in Figure 2.1.

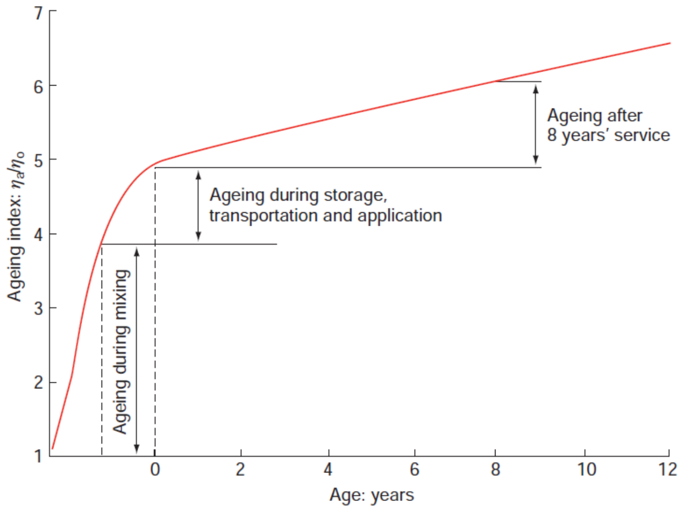


Figure 2.1: Short term and long term aging of bitumen (Robert et al. 2015)

An attempt has been made in the next sections to briefly discuss the chemistry and molecular structure of the bituminous binders and mineral aggregate fillers as a preamble to the discussion on the aging of bituminous binders.

2.2.1 Bitumen

Bitumen is a viscoelastic material and it is majorly manufactured from crude petroleum oil or to a lesser extent from the natural deposits. The elementary analysis of bitumen shows that the chemical composition of bitumen is complex with 82 – 88% of Carbon and 8 – 11% of Hydrogen atoms. The other elements like Sulphur up to 6%, Oxygen up to 1.5% and Nitrogen up to 1% may also be present (Robert et al. 2015).

Molecular structure and fractional composition of bitumen

The organic molecules of bitumen vary widely from highly aromatic, highly polar structures containing varying quantities of heteroatoms (such as nitrogen, oxygen and sulphur) to non-polar, non-aromatic hydrocarbons (Petersen 1984).

It is very difficult to analyze bitumen chemically as number of molecules with different structures are very large. The chemical analysis is also difficult because the precise composition of bitumen is affected by the parent crude oil, method of distillation etc. Hence, instead of separating and identifying all the molecules in bitumen, the chemists have divided the bitumen based on molecular size, polarity and chemical reactivity, into groups (Petersen 1984). The fractional separation methods developed by Corbett (1969) is widely used for defining bitumen composition.

In the process developed by Corbett (1969), when n-heptane is added to bitumen, the asphaltenes are obtained first as they are insoluble in n-heptane and remaining fractions are commonly called maltenes. The maltenes are further divided based on their polarity, into saturates, aromatics and resins. These four fractions are explained further for better understanding.

Asphaltenes: These are insoluble fraction of bitumen in n-heptane which are black or brown amorphous solids. The asphaltenes contain carbon, hydrogen, sulphur, nitrogen and oxygen. These impose a significant effect on rheological properties of binder. The growth in the asphaltene content will lead to a very hard bitumen with higher softening point, reduced penetration and consequently greater viscosity. Asphaltenes constitutes 5 - 25 % of the bitumen (Robert et al. 2015).

Resins: These are soluble fraction of bitumen in n-heptane. Resins are dark brown semi solid or solid fractions with very high polarity, which results in adhesive properties. Resins constitutes around twenty percent of the bitumen (Robert et al. 2015).

Aromatics: These are also a soluble fraction of bitumen in n-heptane. They are usually dark brown viscous liquids and are non-polar in nature. They are the major portion of dispersion medium for asphaltenes and they contain about 40 - 65 % of the total bitumen (Robert et al. 2015).

Saturates: These are also a soluble fraction of bitumen in n-heptane. They are usually viscous liquids and are non-polar in nature. Molecular weight of saturates are similar to those of aromatics. Saturates constitutes around 5 - 20% of the total bitumen (Robert et al. 2015).

2.2.2 Mineral aggregates and fillers

The mineral aggregates and fillers constitute the largest portion of bituminous concrete mixture and play a role in the aging phenomenon of bituminous concrete as a whole. The aging of bitumen is affected by aggregates in following ways (Wu 2014).

- The minerals present on the aggregate surface may increase bitumen oxidation.
- The absorption of bitumen fraction by aggregate may lead to less dispersed binder content, thus promoting the aging

The aggregates are made up of different minerals in which the silicates are the most important and largest. It is estimated that silicates constitute around 90% of earth crust. The carbonate minerals are also important because the limestone ($CaCO_3$), also used as paving aggregates, is made up of carbonates.

The mineral fillers are the special fraction of aggregates which passes the 75 μm sieve. Initially filler is thought to be a part of aggregate system which fills the voids between the aggregates particles. But the recent studies (Recasens et al. 2005, Iwanski and Grzegorz 2013, Lesueur et al. 2016, and Rasouli et al. 2018) have shown that the fillers with their fineness and surface characteristics play a role, more than just being a void filler.

2.2.3 Factors affecting aging

The aging of bitumen is influenced by numerous factors. Traxler (1963) has identified the factors which lead to aging there by affecting the rheological, chemical and adhesion properties of bitumen. These factors are tabulated below in Table 2.1.

Table 2.1: Factors affecting bitumen aging (Robert et al. 2015)

S. No	Factors	Affected by			
		Time	Heat	Sunlight	Oxygen
1	Oxidation (in dark)				Yes
2	Volatilization			Yes	Yes
3	Polymerization				
4	Steric or physical			Yes	Yes
5	Exudation of oils	YES	YES	Yes	
6	Action by water			Yes	
7	Absorption by solid				
8	Chemical reactions				
9	Microbiological deterioration				

Among all the listed factors, oxidation plays a very crucial role in aging of bitumen. According to Petersen (1984) the most important property of bitumen to make it durable is its ability to resist the chemical changes, when in service.

Petersen (1984) has reported the following as the factors that cause aging in bitumen during the mixing process and service life.

- Oxidation caused due to the interaction between oxygen and bitumen. The rate of oxidation depends on factors like type of bitumen and the availability of oxygen.

- Volatilization occurs due to the evaporation of volatile fractions from bitumen and it is caused by the temperature exposure. This occurs mainly during the mixing stage.
- Polymerization: This occurs when same type of molecules combines to form larger molecules, which in turn results in progressive hardening. This property also depends on temperature.
- Steric hardening: This occurs at room temperature with the progression of time. This phenomenon involves the molecular reorganization of bitumen affecting the asphaltene fractions. The steric hardening may be reversible due to the temperature fluctuations.
- Syneresis and separation: This is also an oxidation reaction which causes thin oily liquids, observed on the surface of the bitumen film. Separating this oily film causes hardening. Separation may also occur due to the absorption of bitumen by aggregates.

In addition to the factors mentioned above, the rate of aging depends on the following factors as observed by Petersen (1984):

- The thickness of the bitumen film on the aggregates (Faster aging with less film thickness and vice versa).
- The air void content in the bituminous concrete (high air void content allows more oxygen and UV ray penetration, thus accelerating the aging).

2.2.4 Methods for delaying bitumen aging

Substances such as rubber powder and polythene are added to bitumen as modifiers. When added they will absorb the light oil, thereby reducing the free radical content, which in turn will improve the anti-aging properties. The crumb rubber modified bitumen is reported to have shown resistance to the short term aging of bitumen (Ali 2013). Hydrated lime is also found to be effective in delaying aging (Recasens et al. 2005).

As discussed earlier, oxidation is one of the main factors which increases the rate of aging. The addition of antioxidant and anti-ozone agents will highly enhance the bitumen anti-aging properties. Some types of rejuvenators like waste oil, castor oil which are oil based are employed to reduce the viscosity and hardness and to delay the aging process (Yu et al. 2009).

The warm mix asphalt (WMA) technology is more popular in the industry to reduce the aging in bituminous mixtures. The reduced aging in WMA bitumen is generally attributed to the reduced oxidation, volatilization, as the mixing and laying temperatures are lowered (Jie et al. 2013).

2.3 Methods for simulation of aging in laboratory

Simulating the short term and long term aging of bitumen and bituminous concrete has always been aspect well researched topic and many researchers have attempted with different accelerated aging techniques. Following techniques of aging were suggested by Anderson (1994) for bitumen individually or in combination with aggregates in bituminous concrete mixtures.

- Heating the bitumen at elevated temperatures
- Decreasing the bitumen film thickness, thus increasing the bitumen surface area being exposed
- Increasing the air / oxygen supply to bitumen samples
- Application of pressure to accelerate diffusion of oxygen

2.3.1 Aging tests for bitumen

Over the years, researchers have developed numerous tests for simulating the accelerated laboratory aging of bitumen binder. Almost all the tests used thin binder film subjected to heating in ovens to increase the rate of aging in bitumen binders. All these tests depend on the extended heating procedures, which cause volatilization.

Among the tests which employ extended heating procedure, few are standardized and are extensively used for controlling the short term aging. The most widely used standardized tests for simulating the short term aging or aging that occurs during mixing are thin film oven test (TFOT), rotating thin film oven test (RTFOT) and the rotating flask test (RFT). For simulating the long term aging of bitumen binder, pressure aging vessel test (PAV) and the rotating cylinder aging test (RCAT) are widely used. These tests are briefly discussed in the following sections.

2.3.1.1 Thin Film Oven Test (TFOT)

The thin film oven test is used for measuring the collective effects of air and heat on a thin film of a bitumen binder. TFOT procedure is standardized by EN 12607-2 and ASTM D 1754. This test was first developed by Wellborn et al. (1940) to differentiate between different types of bitumen with different hardening and volatility characteristics. The main purpose of TFOT is to reproduce the extent of aging that occurs to bitumen while mixing with aggregates in the mixing or production plant. Thin Film Oven Apparatus is shown in Figure 2.2 for reference.

The test procedure consists of taking a 50 ml bitumen sample in a 140 mm diameter flat container which results in a thin film of thickness 3.2 mm. For each test, at least two, if needed more, of the above-mentioned flat containers are placed on a rotating shelf which rotates at 5 to 6 rpm in an oven at 163 º C for 5 hours. The effect of aging which occurred during testing is determined by the change in mass of bitumen or as a change in bitumen properties like softening point, penetration or viscosity before and after testing.

The main drawback of this test is that, only the top skin of bitumen sample is aged due to volatile loss. This is primarily because of the thick film sample used, with large volume of bitumen exposed surface area and the bitumen sample is not agitated or moved to make the aging more uniform throughout the sample. To overcome this drawback, modified thin film oven test was developed, in which the binder film is decreased from 3.2mm to 100 µm (Airey 2003).

Figure 2.2: Thin Film Oven Test Apparatus (Controls-group, 2019)

2.3.1.2 Rolling Thin Film Oven Test (RTFOT)

One of the basic tenets of the Superpave Performance Grade (PG) binder specification is that tests should be as closely simulated as possible with the field performance. Superpave PG binder specification calls for short term aged asphalt binders to be investigated at elevated temperatures to find rutting and fatigue behavior. RTFOT provides a measure of the quantum of volatiles lost during the aging phenomenon.

This test is developed by modifying the TFOT test to overcome the issue of non-uniform aging that occur in the test sample. The RTFOT is one of the most widely used tests, standardized through AASHTO T 240 and ASTM D 2872 codes. It was developed by the Division of Highways, State of California Department of Public Works (Hveem et al. 1963). RTFOT is used as a standard test under the SHRP binder specification to simulate short term aging that occurs in hot-mix asphalt plant. Test setup is presented through Figure 2.3 for reference.

In RTFOT, Fresh bitumen sample weighing 35 g each is placed in 8 cylindrical jars and all the jars are placed in a carousel fitted inside a special oven. The oven is then heated to 163 ° C and the carrousel is rotated at 15 rpm for 85 minutes. Flow of air is fixed at 4000 ml/min. The carousel rotation continuously exposes new asphalt binder to the heat and air flow and slowly mixes each of the samples.

RTFOT do not simulate exact conditions found in practice. However, the results obtained from this test correlate very closely with those of conventional batch mixer. The effect of aging is determined by the change in mass of bitumen or as a change in bitumen properties like softening point, penetration or viscosity before and after testing. However, this test is not applicable to the binders which are modified or to those where the viscosity is too high. Minor modifications have been attempted by researchers like extending the duration of testing, replacing air with nitrogen gas etc. (Bahia et al. 1998).

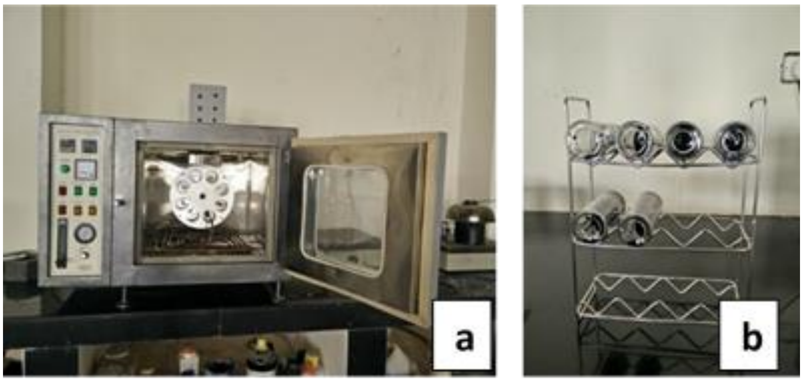


Figure 2.3: (a) Rotating Thin Film Oven Test Apparatus
(b) Sample holder stand

2.2.1.3 Modified Rolling Thin Film Oven Test (MRTFOT)

When additives are added to bitumen binders, the viscosity of the bitumen will get effected. The viscosity of the bitumen may increase or decrease depending on the additive being used. If the viscosity is increased, the binder will not roll inside the glass container, thus making the test ineffective. Likewise, if the viscosity of the binder is reduced, the bitumen will roll out of the glass containers during RTFOT test. To avoid these problems Bahia et al. (1998) proposed Modified Rolling Thin Film Oven Test (MRTFOT)

MRTFOT is similar to RTFOT except that steel rods of 127mm long and 4.6 mm are placed inside the containers during the aging process. These steel rods will cause shearing forces to sweep the bitumen into thin layers, followed by exposing the films to heat and air, thus facilitating aging. According to Bahia et al. (1998), the rods used in this test do not have any effect on the conventional bitumen. Moreover, the metal rods do not address the problem of spillage of bitumen binder when low viscous bitumen is used.

To overcome the problem of roll out of bitumen from glass containers the Rapid Recovery Test (RRT) uses screws to draw the bitumen sample to the back of the glass container during the testing in RTFOT.

2.3.1.4 German Rotating Flask Test (GRFT)

This test was proposed as a replacement to TFOT and RTFOT (Zupanick, M., Baselice, V., 1997). The German Rotating Flask Test (GRFT), which is shown in Figure 2.4, was developed to facilitate the short term aging of modified binders. The main advantage of this aging method is that, its dynamic conditioning test method, effectively avoids the formation of skin and segregation of polymers (which are present in bitumen) that occurs during static type of aging techniques like TFOT.

For aging the binder, 100 g of bitumen is placed in a spherical flask which is tilted and submerged in an oil bath which is heated at 165 ° C. The spherical flask is rotated at a speed of 20 rpm with 500 cc/min of air passing through the flask for 150 min. The rotation of spherical flask causes the sample to turn over during the process and thus prevents the formation of skin. This test offers the control over the volume of gas that passes over the binder as the test is conducted in a closed vessel. The other advantages of this test are that the radiant heat problems are reduced, and the sample is heated quickly as the test is conducted in oil bath. As the test is carried in a closed flask, it facilitates the collection of volatilized components also.

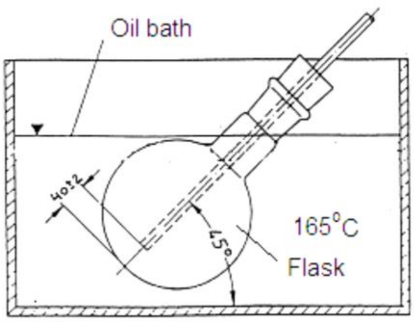


Figure 2.4: German rotating flask (Zupanick, M., Baselice, V., 1997)

2.3.1.5 Rotating Cylinder Aging Test (RCAT)

The high temperatures that the specimens are subjected to, in standard aging tests like TFOT and RTFOT, can simulate the short term aging closely. However, these tests are not suitable for simulating the field or in situ aging. In this context, Belgium Road Research Center (BRRC), based on the principal of theoretical kinetic approach to bitumen aging (Verhasselt, 1991) have developed the accelerated aging apparatus to simulate the long term aging.

The apparatus, shown in Figure 2.5, consists of a large cylinder with an internal diameter of 124 mm and 300 mm long. 500 g of bitumen can be placed in the cylinder at once. After introducing the bitumen binder in the cylinder, a steel roller of 34 mm in diameter and 296 mm in length is set in the cylinder. This cylinder with bitumen sample is introduced into a frame that rotates the bitumen in the container at one revolution per minute while flowing oxygen at 4 to 5 liters/hour. The test is carried out with temperatures ranging between 70 and 110 º C. The rotating cylinder along with the roller distributes the bitumen binder into a film of 2 mm thickness on the inner surface of the container. Approximately 20 to 25 grams of bitumen is recovered at discrete intervals from the container for further testing. As the quantity of bitumen used is large in the processes, numerous evaluations and investigations can be made on the progressive physical properties and bitumen chemistry.

Choquet (1993) and Verhasselt (1997) found correlation between the laboratory aging done with rotating cylinder aging test (RCAT) and in-service aging in the field for dense mixtures. Francken et al. (1997) identified that increased aging times more than 240 hours were required to emulate field aging when it comes to aging of porous mixtures.

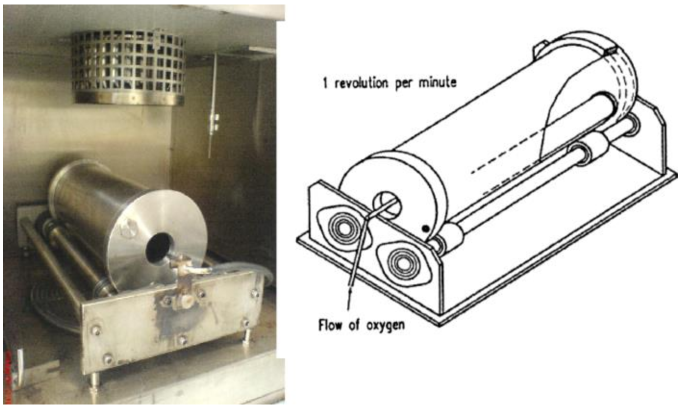


Figure 2.5: Rotating Cylinder Aging Test apparatus (Verhasselt 2000)

2.3.1.6 Pressure Aging Vessel (PAV)

To replicate the field aging that occurs during the life of pavement due to oxidation in bitumen binder, the researchers at SHRP developed Pressure Aging Vessel (PAV), which is shown in Figure 2.6. To carry PAV test, the bitumen binder is first short term aged by TFOT or RTFOT method, followed by pressure oxidizing the residue collected in a PAV. The procedure involves pouring 50 g of bitumen into a preheated pan of 140 mm diameter. The thickness of the binder that is spread on the pan will be around 3.2 mm. The pan is placed in a shelf rack which can hold 10 such pans. The temperature of the pressure aging vessel is maintained between 85º C to 110 º C and at a pressure of 2.07 MPa during the aging process that will be done for 20 hours. The aging temperatures that are below 100 º C are recommended to achieve the physical and chemical changes that occur in field aging.

The PAV is modified using lower temperature of 85 º C for 65 hours under 2.07 MPa air pressure and the resulting equipment is called as High-Pressure Aging Test (HiPAT). These modifications are done to simulate the real time field pavement temperatures more closely. Moreover, the temperatures that are employed in the PAV test are very high so that the modified binders are altered when they are PAV aged and binders become unrepresentative to that of field. Hayton et al. (1999) found that HiPAT process is harsher when compared with the natural field aging for dense asphalt mixture pavement of 10 years old service life.

The major drawback of the PAV test is that it is a static test and the oxygen is not diffused throughout the sample causing the non-homogeneous mixture leading to the different aging at surface and inside of the sample (Verhasselt 2002). A summary of various bituminous aging methods is presented in Table 2.2

Figure 2.6: Pressure Aging Vessel (PAV) (Pavement Interactive 2019)

Table 2.2: Summary of different aging tests (Airey 2003)

Aging Test	Temp (° C)	Time (hrs.)	Wt. (g)	Film (mm)	Limitations
Thin film oven test, TFOT	163	5	50	3.2	Only the top skin of bitumen sample is aged
Rolling thin film oven test, RTFOT	163	1.25	35	1.25	Do not simulate exact field conditions
Modified thin film oven test	163	24	-	0.1	Spillage of bitumen binder when low viscous bitumen is used
German rotating flask test, GRFT	165	2.5	100	-	Difficult set up and less sample
Rotating cylinder aging test, RCAT	70-110	144	500	2	Increased aging times
Pressure aging vessel, PAV	90-110	20	50	3.2	Oxygen is not diffused throughout the sample

2.3.2 Aging tests for bituminous concrete mixtures

The aging phenomenon which is observed in the field is influenced by the nature of aggregates used. Hence, it is more practical and relevant when the bituminous concrete mixtures are tested for aging. As in the case of bitumen aging, a wide variety of tests exist for laboratory aging of bituminous concrete mixtures. The effect of aging is estimated by artificially aging the mixture and finding the change in the material parameters like stiffness, strength etc. These aging methods are mainly divided into four categories (Airey 2003):

- Oxidation tests
- Extended heating procedures
- Ultraviolet or Infrared treatment
- Steric hardening.

The laboratory aging procedure of bituminous mixtures were further classified by Kim et al., (2013) for National Cooperative Highway Research Program (NCHRP) as:

- State of material used i.e., compacted specimen or loose mix
- Pressure level i.e., oven aging or pressurized aging.

2.3.2.1 Loose mixture aging

The aging of loose mixture is less common when compared with the aging of compacted specimens, although some studies have recommended the aging of loose mix as it represents the aging of bitumen pavements more accurately (Bergh 2012, Mollenhauer et al. 2012). The main advantages of loose mixture aging are:

- Uniform aging throughout the sample as the circulation of air and heat can be done inside the loose mix.
- Absence of slump after aging, as seen in the aging of compacted specimens.
- The rate of oxidation is higher as a large surface area of binder is exposed to oxygen.

The main limitation of this loose mixture aging is the difficulty involved in the compaction of the mixture into specimens after aging (Reed, 2010). Although the loose mixture aging seems to be advantageous due to homogeneity and efficiency of aging it is not practical as a lot of effort is required to compact the aged loose mixture into specimens. The compacted specimens are required for performance testing purposes. Due to the stiffness present in the loose mixtures after aging, very high compaction forces are required which may cause aggregate structure to degrade and change the mix properties. The difference in aging between loose and compacted mix is shown in Figure 2.7.

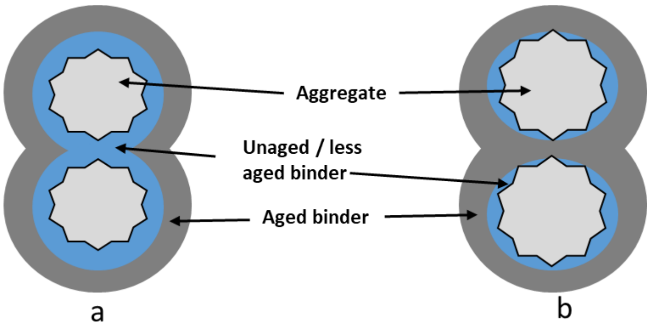


Figure 2.7: (a) Bitumen film on aggregate within the core

(b) Bitumen film on aggregate in the loose mix

(Redrawn based on concept explained by Reed (2010))

The loose mix compaction into a specimen is problematic as a very stiff and hard film of aged bitumen will cause less cohesion which may affect the performance test results (Reed 2010). One method to overcome this problem of compaction is to compact the aged loose mixture at equi-viscous temperatures depending on the viscosity of the mix which is short term aged (Bell 1994). This process requires very high temperature of around 188 ° C for the long term aged sample to get the identical viscosity as that of reference viscosity of short term aged material. These elevated temperatures, if employed, may cause practicality and emission problems during testing in the laboratory. A summary of aging procedures that are usually employed on loose bituminous mixtures is presented in Table 2.3. The tests that are used for aging of loose mixture are presented in the table below (Kim et al., 2013).

Table 2.3: Aging procedures for loose bituminous mixtures (Kim et al., 2013)

Long term aging Procedure	**Equipment**	**Temp. (° C)**	**Time**
Gooswilligen, 1989	Oven	160	16 hrs.
Such et al., 1997	Oven	100	24 hrs.
Read et al., Shell, 2003	Oven	80	7 days
De la Roche et al., RILEM TG 5, 2009	Oven	85	7-9 days
Pierard et al., BRRC, 2009	Oven	60	14 days
Mollenhauer et al., 2011	PAV	90	20 hrs.
Van den Bergh, 2011	Oven	85, 90	7 days

2.3.2.2 Compacted specimen aging

The aging of compacted specimens facilitates the evaluating the performance of bituminous concrete mixtures throughout the useful life of bituminous pavements. AASHTO R30 procedure for compacted specimen aging was standardized during the year 2002. The procedure involves subjecting the bitumen mixture to a short term aging in a preheated oven at 135° C for four continuous hours, to simulate the aging that occurs while mixing and laying. Then, this aged mixture from oven is compacted into specimens. These compacted specimens are placed in an oven for long term aging which is carried at around 85° C for 120 hours to indicate aging that occurs in the field during a specified period of 5 years (Kim et al., 2015).

The aging of compacted bituminous mixtures by AASHTO R30 has a few limitations like:

- The test specifies only one aging temperature to represent a wide range of temperatures seen in the field.
- The simulation cannot be done for periods more than ten years.
- Although it is established that the air void percentage of the mixture plays a major role in the aging of bituminous mixtures, the void content is not taken into consideration.
- It is observed that the temperature and the time period used in this test is not enough to simulate the extent of aging occurring in the field,

Apart from aging of compacted bituminous mixtures by AASHTO R30, many other aging procedures were developed and proposed for aging of compacted mixture specimens. These procedures vary with respect to the duration and temperature of testing. Different techniques that are used for aging of compacted bitumen specimens are summarized and presented in Table 2.4.

Table 2.4: Aging procedures for compacted bitumen specimens (Kim et al. 2013)

Long term aging Procedure	Equipment	Temp. (° C)	Time
Hveem et al., 1963	Oven	60	1000 hrs.
Mugler, 1970	Oven	163	5 hrs.
Tia et al, 1988	Oven	60	90 days
Von Quintus et al., 1992	Oven	60 - 107	7 days
Bell et al., AASHTO R30-02, 1994	Oven	85	5 days
Martin et al., 2003	PAV	70 - 80	7 days
Van den Bergh, 2011	Oven	110 - 120	16 hrs.

2.3.2.3 Oven aging

Oxidation is identified as the major cause of aging and to replicate oxidative aging of bituminous mixtures in the lab, Long term oven aging (LTOA) is used. The current standard method for aging bituminous mixtures AASHTO R30, specifies the conditioning of compacted bitumen concrete samples in an oven at 85° C for a continuous period of five days.

The SHRP researchers conducted an extensive study on the long term aging procedures for bituminous mixtures. Long term oven aging (LTOA) was carried at both 85° C and 100° C and the results indicated that the aging was similar. The level of aging was reached more quickly when 100° C was used, but the greater specimen to specimen variability was observed. Hence, the temperature of 85 ° C was finalized by the researchers for Long term oven aging (Bell 1989).

The principle advantage of oven aging is the ease with which the aging is achieved. Also, the easy availability of these ovens is considered a definite advantage. In addition, large quantities of mixtures can be subjected to aging at a time. However, these ovens need more time to simulate the oxidation levels to simulate field conditions, more closely. These ovens differ with respect to their function of air drafting and this variability can influence the aging process of bitumen concrete mixtures.

2.3.2.4 Pressure aging

Pressure along with air can be employed to enhance the rate of oxidation in bituminous concrete mixture specimens as an alternative to oven aging. The pressure aging of bituminous mixtures was tried by several researchers for both loose and compacted mixtures. The main advantage of pressure aging is that it is reliable than oven aging and the instrument variability is very less between the laboratories when compared with oven aging (Kim et al. 2013).

Although the pressure aging is widely accepted it has a few disadvantages like only a small quantity of material can be aged in one cycle. Hence, it takes lot of time to arrive at the quantities that are required to test for aging. One more area for concern in pressure aging is that of integrity of compacted samples.

It is seen in the previous sections that a significant amount of work is done to understand the aging process of bitumen binder, relatively less work is done to develop accelerated lab aging procedures for bituminous mixtures. Moreover, the validation of laboratory aging methods with that of field measurements is very limited. The climatic conditions, depths etc., are not considered in the laboratory aging methods that are developed for aging of bituminous mixtures.

2.4 Literature review of the research on bitumen aging

The studies on asphalt aging have begun in 1903 by Dow. A. W (Vargas, X., Reyes, F., 2010). Since then, the understanding of asphalt / bitumen aging has grown. With the advent of state of the art instruments, which can simulate complex field conditions in the lab, and also by using very powerful microscopic technologies like Atomic Force Microscopy (AFM), Field Emission Scanning Electron Microscopy (FE SEM); the researchers are now in a position to understand the aging phenomenon more clearly than ever. The contributions by different researchers in the areas including the aging of bitumen and bituminous concrete alongside the micro structural investigations on aged binders is summarized and presented in the following two main sections with a view to identify the gap for further investigations.

2.4.1 Literature review on bitumen aging

The focus of the current research activity is the simulation of field aging of bituminous concrete with accelerated laboratory aging protocols, as closely as possible. Hence, a detailed literature review was carried out with regard to aging investigations carried out by different researchers and a summary of the major observations are presented in this section.

Stephens, J.E., Santosa, W. (1992) have aged the asphalt mixture samples in oven at 60 ° C and 7 kPa air pressure to find that the critical pavement voids are in between 9% and 13%. Kandhal and Chakraborty. (1996) have found that the film thickness of 9 to 10 microns is optimum and the less thickness causes the accelerated aging. Harvey, J., Tsai, B.W. (1997), Huang et al. (2012) and Cheolmin, B. et al. (2012) indicated that after the long term aging of asphalt in oven for 6 days, the stiffness of asphalt was found to be increasing. It was documented that the pavement fatigue life is more dependent on the aggregate and asphalt types and air void content. Lamontagne et al. (2001) analyzed three different types of bitumen aging studies and opined that cell oxidation technique can reproduce the field conditions closely.

Li et al. (2006), Valtorta et al. (2007), Woo et al. (2008), Jemere (2010) and Jing et al. (2019), after testing for rheological and chemical properties have found that the top layer aged more than the lower layers in an asphalt concrete layer. It was also found that the temperature had more profound effect in oxidation process than pressure. Wang et al. (2019) analyzed and characterized the aging of bitumen from different structural layers of pavements from their microstructures and compositions. It was seen that the degree of aging of bitumen in different layers of the pavement structure in service was different, but they conform to the same order of aging degree, which was, upper layer > middle layer > bottom layer.

Ongel et al. (2014) have reported that the presence of oxygen is necessary for aging because no aging was observed when nitrogen was used in the sample, thus establishing that aging is solely caused by oxidation. It was observed that temperature had direct effect on aging to such an extent that the oxidation got doubled with 10° C increase of temperature. Hofko et al. (2015) have tried an alternative approach for aging. During this study, it was found that the other atmospheric gases like ozone, nitric oxide etc., which are in low concentration, were observed to be causing the long term aging. The water-soluble oxidants were also found to be enhancing the aging as they penetrate binder and base layers.

Dessouky et al. (2015) have experimented the influence of antioxidants on bitumen rheology along with mechanical performance. The elastic behavior of the asphalt after aging was observed to have increased when antioxidants were used, as observed from temperature and frequency sweep testing. Xu et al. (2017) have simulated the influence of oxidative aging on thermodynamic properties of bitumen using Molecular dynamics simulation. It was concluded that by replacing hydrogen atoms with oxygen atoms in asphalt, the oxidative aging can be simulated satisfactorily. Nazari et al. (2018) have experimented with the inorganic nanoparticles for increasing the aging resistance and concluded that both SEM and XRD investigations are adequate for analyzing microstructures of modified binders. The TiO2 and CaCO3 nanoparticles were found to be good antioxidants and increased the fatigue life as complemented by the results of Time Sweep and Linear Amplitude Sweep tests.

Button (1988) examined the effect of additives like SBS, EVA, Polyethylene and latex in highway construction and presented that the first three additives resisted the aging better than latex modified asphalt. Lu et al. (1998) used Gel Permeation Chromatography (GPC), Dynamic Mechanical Analysis (DMA) and Fourier Transform Infrared (FT-IR) spectroscopy to investigate the aging of SBS Polymer modified bitumen. It was seen that the aging increases the sulphoxides and carbonyl compounds and the SBS acts as antioxidant.

Recasens et al. (2005) evaluated the effect of fillers i.e. calcium carbonate and hydrated lime on the phenomenon of aging and concluded that the fillers are effective in preventing aging and they should be added on basis of volume rather than weight. Lesueur et al. (2016) have developed a simple test method to assess the influence of mineral fillers on aging. It was observed that hydrated lime had significantly reduced the aging effects, but limestone filler failed to do so. Rasouli et al. (2018) evaluated fatigue behaviour by using hydrated lime in asphalt mixtures and concluded that the replacing the aggregate filler with hydrated lime improves the flexural stiffness and decreases the phase angle. It is found that the mixtures using hydrated lime had longer fatigue life when analyzed. Morian et al. (2011) in their study had examined the effect of aggregate sources, mixture characteristics on binder aging and stiffness. It was reported that the mixture characteristics clearly influence the oxidative aging phenomenon.

Ahmed et al. (2012), Gandhi et al. (2010) and Jie et al. (2013) have examined effects of warm mix additives and dispersants on bitumen binder rheological properties and aging properties. It was observed that additives had very positive effect in reducing the chemical and physical hardening tendencies. The warm mix additives improved moisture susceptibility of bituminous mixes. It was found that addition of Sasobit changed the viscosity of the binder thus facilitating the reduction of mixing temperature substantially. The rutting resistance got improved while the fatigue cracking and thermal cracking resistances got compromised marginally, when compared with the neat binder.

Yin et al. (2015) have investigated short term aging and noted that the short term oven aging of two hours at 116° C for WMA and of two hours at 135 ° C for HMA is adequate for reproducing the asphalt absorption during production at plant. Xiao et al. (2015) observed that the binder source and grade significantly affect the Ultraviolet and PAV aging procedures. The warm mix additives have no influence on phase angle and G* values of binder after aging. Zhang et al. (2017) have evaluated the long term field aging of mixtures with WMA. It was found that aging rate of the binder may be nonlinear or linear based on the type of the binder. The aging was found to have profound impact on wheel path top down cracking.

Zhang et al. (2010) have evaluated the effects of aging on rheological properties of SBS and sulfur modified asphalts and confirmed that the oxidative aging has major influence on the rheological behavior of the asphalts that are modified. Airey et al. (2011) have indicated that the polymer modification of the asphalt binder will increase the elastic response, complex modulus and viscosity, thereby, enhancing the rheological properties.

Fátima et al., (2013), Iwanski and Grzegorz (2013) found that the hydrated lime, poly phosphoric acid and SBS binders showed higher photo degradation resistance when compared with conventional binders in infrared spectroscopy results. It was observed that the aging process had slowed down as hydrated lime facilitated greater adhesion and durability. Hydrated lime acts as antioxidant and decreases the stiffness of the asphalt.

Pan et al. (2014) in their investigations on using graphite as anti-aging material, documented that graphite had anti-aging properties which was seen by physical and rheological characteristics of neat and graphite modified aged binders. Amri et al. (2015) have analyzed the influence of sodium borohydride as reducing agent in resisting the aging and its impact on chemical structure. It was established that reduced bitumen was relatively uninfluenced to the formation of sulfoxide and carbonyl groups.

Lu and Isacsson (2002) studied the effects of aging on the chemistry and rheology of bitumen and opined that due to the chemical changes caused by aging, the complex modulus had increased while the phase angle got reduced. Said et al. (2005) have presented the effects of aging on mechanical properties of bituminous mixtures. It was reported that the hardening of asphalt occurs rapidly in the initial life of the roads and both the creep and stiffness modulus have shown diurnal variations during the first year after laying of the pavement.

Claine P. J. et al. (2011) have compared the previous research findings regarding the oxidation mechanisms with their own results. They reported that sulfoxides and ketones are formed in long term aging and the alcohols are responsible for age hardening and viscosity increase during oxidation. Feng et al. (2012) and Qin et al. (2013), used colloidal elements like saturates, resins, aromatics and asphaltenes to evaluate the relation between the aging of bitumen and colloidal chemistry. They have found that the aging had major impact on the quantities of aromatic and asphaltene components. Aromatic components are converted into asphaltenes which can be soluble in toluene. The aromatic, sulfoxide and carbonyl functional groups are increased by field aging.

Poulikakos et al. (2014) and Menapace et al. (2017) have used FT-IR spectroscopy, XPS and Atomic force microscopy to investigate the effects of aging on chemical and micro structural properties of recycled mixtures. It was observed that the aging of binders caused the increase of sulfoxide and carbonyl components and the analysis of chemical changes showed severe chemical changes when compared with standard short term and long term aging methods. Hou et al. (2018) and Hu et al. (2018) used spectrophotometry for analyzing the aging and proposed that the aging in essence means, decrease in maltene content with the corresponding increase in asphaltene content. From the FT-IR and rheological investigations, it was found that the aging rate of bitumen is different for different thicknesses and different aging techniques led to different results.

Singh et al. (2018) attempted to determine the reclaimed asphalt pavement (RAP) proportion to achieve the target grade of binder (AC30) by using absolute viscosity and PG methods, and compared with RAP proportion obtained from linear amplitude sweep test (LAS) and multiple stress creep and recovery (MSCR) tests. Zhang et al. (2018) evaluated the applicability of the multiple stress creep and recovery test (MSCR) test on fatigue resistance of asphalt binder and to verify whether the MSCR test could represent mixture fatigue performance. They reported that binder MSCR test at intermediate temperatures showed a similar ranking to the mixture's fatigue test, indicating that the binder MSCR test could be potentially utilized to represent a mixture's fatigue resistance where binder selection is a major concern. Jing et al. (2019) focused on the fatigue, relaxation characteristics, and the linear viscoelastic properties, of aged bitumen. A linear relationship was found between the relaxation aging index and the crossover modulus aging index. This relation can be used to classify bitumen upon its ability to resist crack formation by using DSR frequency sweep tests.

2.4.2 Literature review of the micro structural investigations on bitumen

It is an undeniable fact that the micro structural investigations play a major role in understanding morphological, chemical and functional changes happening in the bituminous concrete mixtures with aging. Many researchers have employed different investigation techniques to explain the aging phenomenon. A detailed literature review was carried out to understand the state of the art in this direction and a summary is presented below for reference.

Herrington et al. (1996), through using FT-IR, observed an increase in the relative carbonyl areas after aging. Lesueur et al. (1999) have established that the hydrated lime can be an efficient and effective multifunctional additive to bitumen by employing nuclear magnetic resonance. It was also seen that the hydrated lime increases the stiffness of the modified bitumen. Chen J.S. et al. (2000) have investigated the difference in aging between the field and lab tests and reported that the increase in sulfoxides had resulted in increase in viscosity. Lu et al. (2002) studied the rheology and chemistry of the bitumen when subjected to aging using the Thin-layer chromatography with flame ionization detection and reported that the formation of sulfoxides and carbonyl compounds due to which the aged bitumens became more solid-like with increased complex modulus and reduced phase angle.

Lamontagne et al. (2001), Mouillet et al. (2008), Wu, s. et al. (2008), Kim et al. (2013), Poulikakos et al. (2014) and Kaya et al. (2020) have investigated the modified bitumen with FT-IR and found that the polymer swelling due to aging was caused by the oxidative aging. The aging makes the bitumen more homogeneous when it is modified with SBS due to polymer degradation. They reported that more sulphoxide and carbonyl groups and relatively lees segments of butadiene were available after long term aging with PAV, thus indicating the oxidation reaction of bituminous binder and degradation of SBS. They also found that the values of the aromatics decreased with SBS addition due to aging. Increase in the carbonyl value and the corresponding sulfoxide absorbance value confirmed the increase in oxidation, thus aging. In AFM images for the samples being observed, the bee-microstructures are absent in aged bitumens.

Puello et al. (2013) have demonstrated that the decomposition of asphaltenes is the primary cause to result in change in weight of the bitumen, through thermo gravimetric analysis (TGA). Qin et al. (2013) have investigated the laboratory aging and field aging and their effect on the bitumen rheology and structure. It was seen that the aging of bitumen impacts the asphaltene and aromatic components without changing the resins and saturates content. It was also observed that the field aging affects the low temperature rheology more when compared with lab aging. Wang et al. (2014) have investigated on a field aged binder and opined that the material type and the depth of pavement affect the aging rate. It was established that the loss of saturates is the main factor which causes the aging at the bottom portion of the surface layer.

Yao et al. (2012) used Scanning Electron Microscopy (SEM) and found that the addition of Nano silica to bitumen has resulted in slight reduction of viscosity of the binder. This lower viscosity results in the lower compaction temperature. The Nano silica modified bitumen had significantly higher dynamic modulus when compared with the reference bitumen. Sobolev et al. (2013) have carried out the microstructural investigations of bitumen binders with fly ash using SEM and found that the fly ash particles induced the crack resisting effect at low temperatures. The rheological investigations also supported the feasibility of using fly ash as a filler for improving the performance of bitumen binders.

Eberhardsteiner et al. (2015) have examined artificially aged bitumen with different asphaltene levels using CR tests in both field aged and lab aged conditions. The rise in asphaltene content had resulted in decreased creep compliance. The micelle like structure enclosed in a contiguous matrix was seen in microstructure of bitumen when FESEM and AFM images were observed. Li et al. (2020) used decompression separation device to separate the five bitumen with various aging stages to evaluate the bitumen compositions and the microstructure during aging. It was seen that light compositions were evolved into the heavy compositions of bitumen. The bitumen having more asphaltenes has little change in colloid index (CI index) after aging. The change rate of surface morphology is positively correlated with that of colloid index.

From the Figure 2.8 it can bee seen that the frequency of research publications, on bitumen and bituminous concrete aging, throughout the years is increasing rapidly.

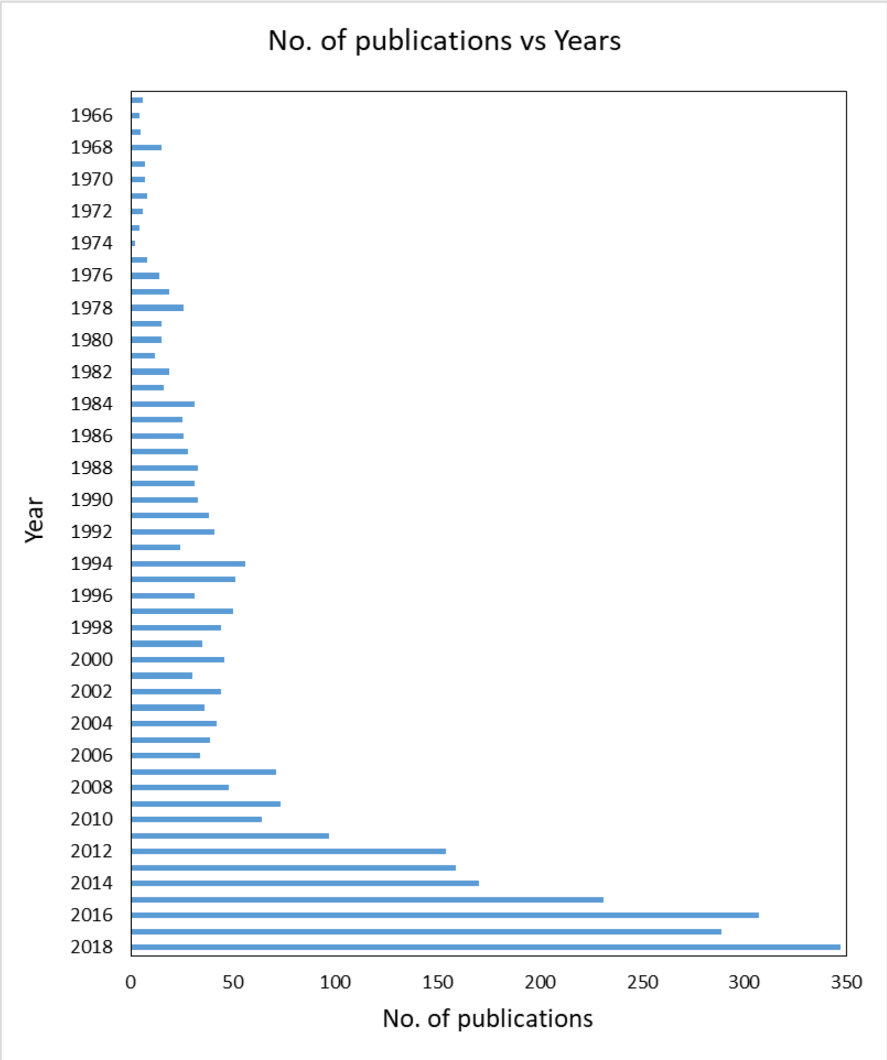


Figure 2.8: Frequency of research publications on bitumen and bituminous concrete aging throughout the years

2.5 Identified research gaps

Extensive literature review on bitumen aging in bituminous concrete layer of a flexible pavement has yielded the following gaps being identified.

- Since long, variety of bitumen modifiers were tried by several researchers with a view to reduce the rate of aging. These modifiers included, but not limited to; polymers, natural rubber, crumb rubber, warm mix additives and rejuvenators. A close examination of these works has provided the clue that the warm mix additives are very effective as they tend to help in substantially reducing the requisite temperature at which the mixture is being prepared, thus resulting in relatively lesser short term aging, consequently leading to improved long term aging resistance. However, studies were very limited with the specific target of investigating the effect of warm mix additives on long term bitumen aging process for Indian conditions.

- Comparative investigations involving the natural field aged bitumen (within bituminous concrete) with those of accelerated laboratory aged bitumen (within bituminous concrete) were not found in literature, for Indian context, in particular.

- The studies on aging of bitumen, supported with different microstructural investigations were found to be very limited and in-adequate to understand the kinetics of progressive aging with the changing microstructure.

- AASHTO R30 oven based aging protocol, though successful in closely simulating the field aging, was developed to suit the typical environmental conditions and construction specifications, prevalent in the United States of America (USA). Attempts to modify this process to suit Indian conditions were found to be missing from the literature. Hence, the lack of availability of oven specifications to simulate the Indian conditions is being identified as a clear gap in the literature.

- Correlating the field and laboratory based aging processes with derived indices from the micro structural investigations was not attempted adequately in the literature. This process is quite promising and expected to lead towards better understanding of the aging process. Hence, this aspect is also being identified as the gap in the current context.

2.6 Research objectives

Long term performance of bituminous concrete majorly depends on the way the bitumen changes its behavior with short term and long term aging phenomena. Understanding, quantifying and simulating the field aging process has always been a major challenge. In the current research activity, a thorough effort had been made in simulating the short term as well as long term aging in the laboratory and investigating the micro structural, rheological and other physical properties of the extracted bitumen residue for it to be compared with the bitumen extracted from the field aged cores. The efficacy of lime as hydraulic filler and anti-stripping agent, and a warm mix additive in the mix in achieving better age resistance is also investigated and reported. To achieve these listed targets, the following objectives have been set for this research study.

1. To standardize the laboratory based long term aging process for bituminous concrete, with a view to simulate the aging happening on a typical Indian road.

2. To investigate the comparative performance of bitumen residue extracted from naturally aged field cores and the artificially aged laboratory specimens with a view to establish the level of artificial aging achieved when compared with the natural aging.

3. To verify the combined efficacy of warm mix additive and hydrated lime in abating long-term aging in bituminous concrete.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

In the previous chapter, a detailed review of literature, pertaining to the chosen research topic, is presented. Specific scope, emerging out of the literature review, was also presented in the previous chapter. Stages, planned to achieve the identified objectives, are explained in detail in this chapter.

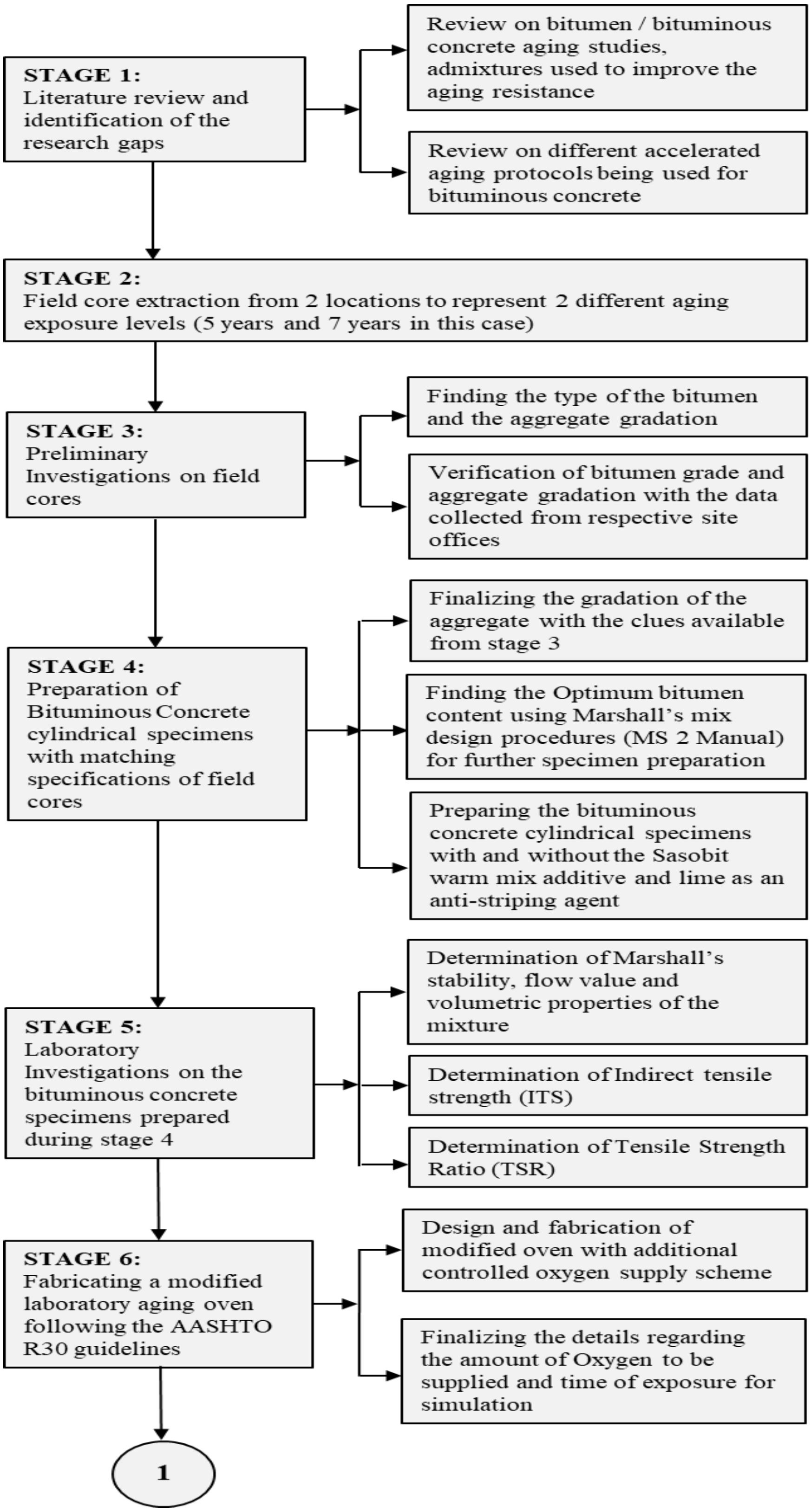
3.2 Stages of research work

The present research work was carried out in 11 stages, as presented in Figure 3.1, is explained in following sections.

3.2.1 Stage 1: Literature review and identification of the research gaps

During the current research, a detailed review of literature was carried out, encompassing all the associated research domains, as listed below.

- Bituminous binder aging
- Bituminous Concrete Aging
- Testing Protocols for aging simulation
- Techniques adopted for countering the aging resistance
- Microstructural investigations on bitumen (both aged and unaged)


STAGE 1:
Literature review and identification of the research gaps
Review on bitumen / bituminous concrete aging studies, admixtures used to improve the aging resistance
Review on different accelerated aging protocols being used for bituminous concrete
STAGE 2:
Field core extraction from 2 locations to represent 2 different aging exposure levels (5 years and 7 years in this case)
STAGE 3:
Preliminary Investigations on field cores
Finding the type of the bitumen and the aggregate gradation
Verification of bitumen grade and aggregate gradation with the data collected from respective site offices
STAGE 4:
Preparation of Bituminous Concrete cylindrical specimens with matching specifications of field cores
Finalizing the gradation of the aggregate with the clues available from stage 3
Finding the Optimum bitumen content using Marshall's mix design procedures (MS 2 Manual) for further specimen preparation
Preparing the bituminous concrete cylindrical specimens with and without the Sasobit warm mix additive and lime as an anti-striping agent
STAGE 5:
Laboratory Investigations on the bituminous concrete specimens prepared during stage 4
Determination of Marshall's stability, flow value and volumetric properties of the mixture
Determination of Indirect tensile strength (ITS)
Determination of Tensile Strength Ratio (TSR)
STAGE 6:
Fabricating a modified laboratory aging oven following the AASHTO R30 guidelines
Design and fabrication of modified oven with additional controlled oxygen supply scheme
Finalizing the details regarding the amount of Oxygen to be supplied and time of exposure for simulation
1

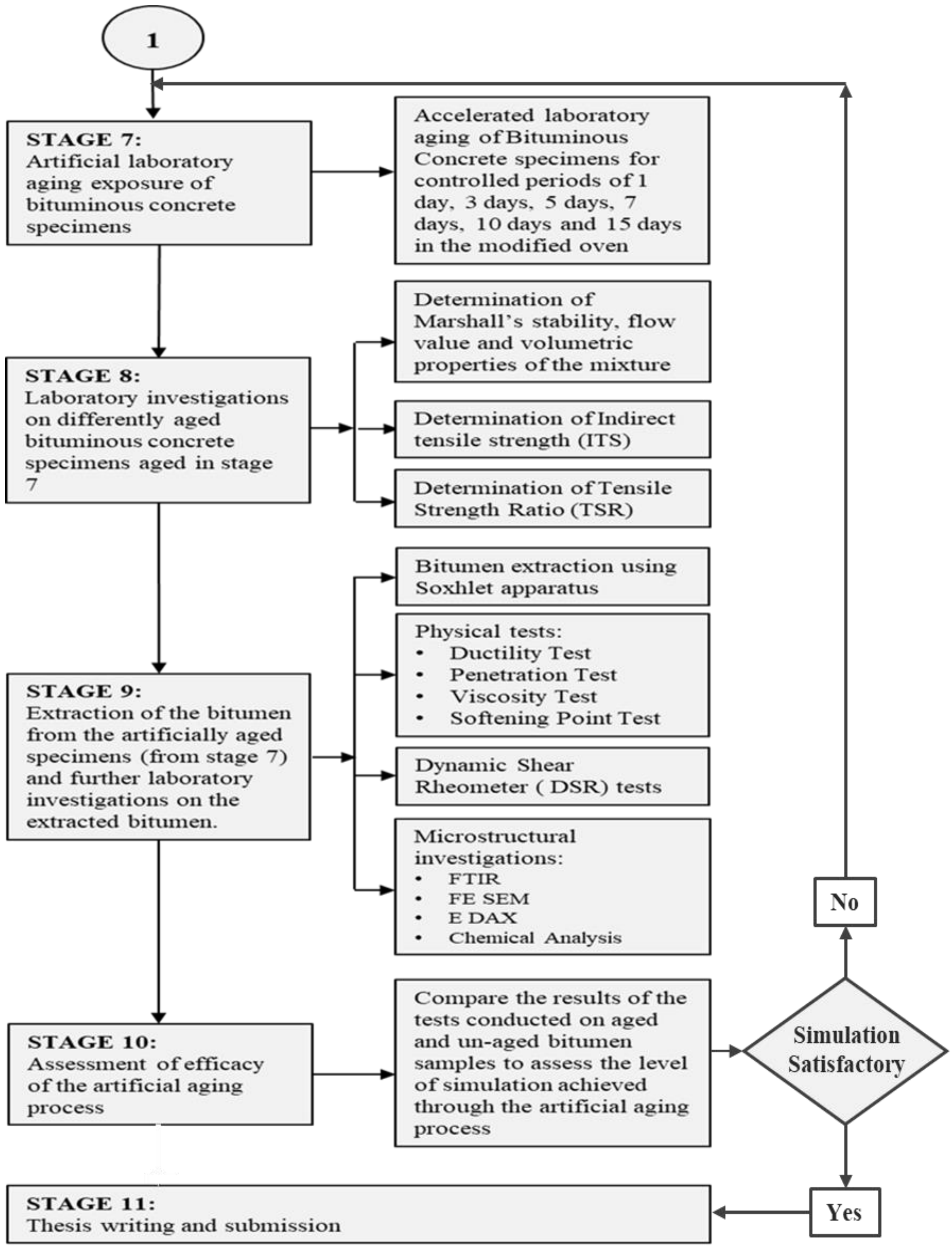


Figure 3.1: Stages involved in the research

3.2.2 Stage 2: Field core extraction from two differently aged field sites

It is proposed to collect the field core specimens from two differently aged pavement sections during this stage. Necessary permissions will be taken from National Highway Authority of India (NHAI) for extracting the field cores. Two differently aged sites, with approximately similar traffic and environmental conditions, will be chosen for making comparative assessment between the two core specimens along with artificially aged lab samples. In addition, it is planned to ensure that the same grade of bitumen and aggregate blend is being employed at both the sites.

3.2.3 Stage 3: Comprehensive laboratory investigations on the field cores

It is a well-established fact that the top 8 to 12 mm thick layer of bituminous concrete surface course undergoes active oxidative aging while the bottom portion literally remains unaged. With this premise, it is proposed to slice the field cores into two parts across the thickness, with the top slice measuring 12 mm while the rest of the sample thickness representing the bottom slice.

Further, bitumen will be extracted separately from both top and bottom slices through reflux process for all the samples collected from the field. The bitumen, extracted from the field cores, will be subjected to variety of investigations to establish the chemical, physical, rheological and morphological changes, which would have happened during the period of real field exposure. In addition, all these investigations will be carried out on virgin bitumen samples too, the results of which will be used as base case.

3.2.4 Stage 4: Preparing bituminous concrete specimens, matching the field specifications

In this stage, bituminous concrete cylindrical specimens will be cast in the laboratory using the modified Marshall's hammer with matching field composition and specifications, observed from both the extracted cores as well as construction records, for further investigations. Optimum binder contents, reported in the construction records, will be verified in the laboratory through preliminary investigations and detailed Marshall's mix design. After confirming the mixture design, it is contemplated make large number of cylindrical specimens for further investigation with the same material blends. This check is necessary to ensure logical comparison between laboratory aged specimens with those of field cores, aged naturally. Additionally, one more set of samples with 2 % of hydrated lime and the optimized quantities of Sasobit warm mix additive will also be cast for establishing the aging resistance offered by the anti-stripping agents and long term advantages derived by using anti stripping agents in terms of reduced aging related failure of the bituminous concrete mixtures.

3.2.5 Stage 5: Laboratory Investigations on the bituminous concrete specimens prepared during stage 4.

Comprehensive laboratory investigations are planned to be carried out to establish the following properties of unaged bituminous concrete specimens.

- Marshall's stability
- Marshall's flow value
- Volumetric properties
- Indirect tensile strength
- Tensile strength ratio
- Rheological properties of binder alone (unaged)
- SARA analysis of the binder alone

3.2.6 Stage 6: Fabricating a modified laboratory aging oven following the AASHTO R30 guidelines.

In this phase, a modified laboratory oven will be fabricated based on "Standard Practice for Mixture Conditioning of Hot Mix Asphalt - AASHTO R 30" guidelines. To simulate the field aging more closely and also to expedite the aging process, it is also proposed to provide controlled oxygen supply to the modified aging oven.

3.2.7 Stage 7: Artificial laboratory aging exposure of bituminous concrete specimens

It is proposed to age the bituminous concrete specimens cast in stage 4 with the modified aging oven, fabricated in stage 6 of this research activity. Care will be taken to simulate both short term as well as long term aging processes together in this stage.

3.2.8 Stage 8: Laboratory investigations on differently aged bituminous concrete specimens (aged in stage 7)

In this phase, it is proposed to test the differently aged bituminous concrete samples to find the properties namely stability, flow, densities, direct tensile strength, tensile strength ratio and fatigue strength. These tests will be conducted on the two sets of unaged and aged bituminous concrete specimens with one set of samples being cast without any additives while the other set, cast with optimized quantities of lime filler and Sasobit warm mix additive.

3.2.9 Stage 9: Extraction of the bitumen from the artificially aged specimens (from stage 7) and further laboratory investigations on the extracted bitumen.

From the aged bituminous concrete specimens, obtained at the end of stage 7, it is proposed to extract bitumen using Reflux binder extractor. This extracted bitumen will be subjected to variety of investigations to establish the chemical, physical, rheological and morphological changes, which would have happened during the laboratory aging exposure. In addition, all these investigations will be carried out on virgin bitumen samples too, the results of which will be used as base case.

3.2.10 Stage 10: Assessment of efficacy of the artificial aging process

Comparative assessment of the properties of artificially aged bitumen samples (observations from 9th stage) with the corresponding properties of field aged bitumen samples (observations from 3rd stage) using appropriate investigations and analysis, will be carried out during this stage.

3.2.11 Stage 11: Thesis writing and submission

In this phase, it is proposed to document the findings in the form of thesis and submit to Departmental Research Committee for further processing.

3.3 Summary

In this chapter, all the stages, proposed during the current research activity, are detailed for comprehensive understanding of the work being carried out. Details of experimental investigations are explained in the forthcoming chapters of this thesis report.

CHAPTER 4

EXPERIMENTAL METHODS AND MATERIALS

4.1 Introduction

Over the years, many techniques were being developed by several researchers to reduce the effect of aging and to improve the mechanical and rheological properties of bitumen, thus enhancing the service life of the pavement. The Shell Bitumen chemical company used Styrene-Butadiene-Styrene (SBS) as an asphalt modifier to improve mechanical properties and rheological behavior (Zhang et al. 2010). It was observed that hydrated lime, as an antioxidant, decreases the rate of aging of bitumen, thus contributing increased level of adhesion and durability to the pavements (Iwanski and Grzegorz 2013). Graphite was also considered as an additive for enhancing the anti-aging properties of bitumen (Pan et al. 2014). Montmorillonite was observed to be improving the physical properties, rheological behavior and storage stability of bitumen (Zhang et al. 2010). Crumb rubber, when added to the bitumen as an additive, enhanced the anti-aging properties of the binder substantially (Ali et al. 2013). On the similar lines, during the recent past, many additives and admixtures are being introduced into the market with a principal target of enhancing the properties of the bitumen.

Sasobit has been a popular choice of additives during the last few years due to many advantages like reduced mixing temperatures, lower air voids, better workability, improved compactability and ease of availability of this product (Jie et al. 2013). Sasobit, with the chemical formula of C_nH_{2n+2}, is generated by gasification of coal and contains a long chain of aliphatic hydrocarbons. This product is a synthetic microcrystalline wax, having finer crystalline structure and longer chain length than the natural asphalt waxes (Ali et al. 2013). The main chain length of hydrocarbons present in Sasobit is of the range 40–115 atoms of carbon, while the natural bitumen paraffin waxes have only 22–45 atoms of carbon (Syroezhko et al. 2011). Due to its proven advantage in reducing the mixing temperature to achieve the workable viscosity of the mix, there by assisting in the reduction of short term aging, Sasobit is considered as an additive during the current investigations.

Hydrated lime was thought to be suitable as a mineral filler to the bituminous concrete mixture. Of late, it was observed to be enhancing the performance of the mixtures' substantially due to the added resistance to chemical aging, moisture damage along with mechanical properties of the mix (Rasouli et al. 2018). It is with this background that hydrated lime is also chosen for this study for investigating its anti-aging properties.

Majority of the works in the past have tried either hydrated lime or Sasobit as an additive. However, investigations with both hydrated lime and Sasobit together as admixtures in bituminous concrete are found to be limited. Validation of the aging phenomenon with micro structural investigations holds great promise and scope. Hence, an attempt towards comparing the effects of aging and micro structural changes in artificially aged specimen with the field aged specimen was made using Fourier Transform Infrared Spectroscopy (FT-IR), Field Emission Scanning Electron Microscope (FE SEM) and Energy Dispersive X-Ray analysis (EDX) analysis. Penetration index, carbonyl index and sulfoxide index were also calculated using established procedures as critical parameters in assessing the level of aging since these functional groups are established as the aging indicators in bitumen (Marsac et al. 2014).

The details of all the investigations, carried out during the current research activity, are presented in the following sections. All the investigations, carried out, are depicted through a flowchart in Figure 4.1, for ready reference.

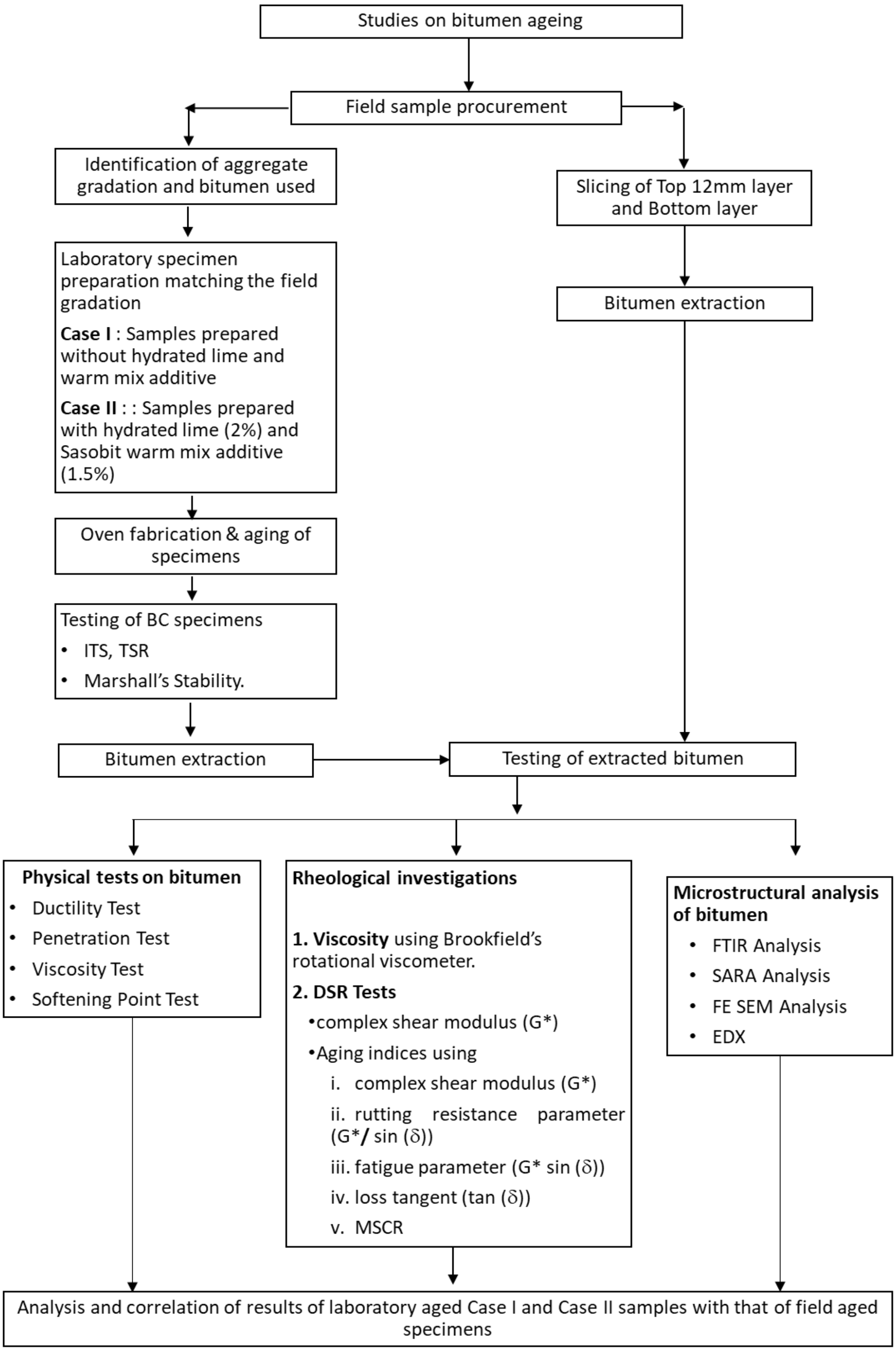


Figure. 4.1 Flow chart of the experimental activity

4.2 Collection of field core specimens of bituminous concrete

Since the current research activity involved simulating the on-field aging process in the laboratory, the collection of cores of bituminous concrete specimens has become mandatory. Two sites were selected for the core collection process based on the following criteria.

- Both the locations shall have similar traffic characteristics and environmental conditions (annual average temperature and rainfall intensity)
- To represent the worst-case scenario, heavily trafficked road sections were chosen as the candidate sections.
- To eliminate the influence of major maintenance activities like overlays on aging, it was verified that the selected locations had only routine maintenance activities like patching and crack filling and never received any major rehabilitation activities during the respective service lives.
- To ensure that the research is leading towards the comprehensive understanding of the on-field aging phenomenon, both the sites are chosen in such a manner that they represent two different aging periods (5 years and 7 years in the present case).
- It was verified that, for both the sites, bitumen grade (VG 30) and bituminous concrete mix (MoRT&H BC Grade I) were same. This is to make the results comparable at a later stage
- To ensure that the core specimens are representative, the cores were collected from both the wheel paths, near the edge of the pavement as well as the central sections.

Based on the above criteria, nine locations each, on two National highways NH 16 and NH 65, were chosen for core collection. At the time of collection of the cores, NH 65 was in service for 5 years while NH 16 was in service for 7 years. The locations, from where the sites are identified are marked on the map and presented in Figure 4.2. Location details, along with traffic level, average temperature and average rainfall are summarized for these two locations and presented in Table 4.1. The gain size distribution of material collected from the field is presented in Table 4.2

Table 4.1: Information of the locations from where cores were extracted (Source – Data was collected from the National Highway site offices)

High way	Location / Section of Core Collected	Service Period	Traffic Level, Commercial Vehicles per Day (CVPD)	Avg. Ambient Temperature (° C)			Pavement Temperature (° C)	Avg. Annual Rainfall, mm
				Min	Max	Annual		
NH 16	Gundugolanu Section - Eluru, AP	7 years	3200	18.8	38.6	28.2	61.37	992
NH 65	Nandigama Section, AP	5 years	3050	17.8	40.7	28.7	63.38	941

Table 4.2: Sieve analysis of field sample

IS Sieve Size (mm)	Percent Passing (%)	
	NH 16	NH 65
26.5	100	100
19	93	95
13.2	72	77
9.5	60	61
4.75	45	43
2.36	35	38
1.18	26	30
0.6	22	22
0.3	14	16
0.15	6	6
0.075	4	5

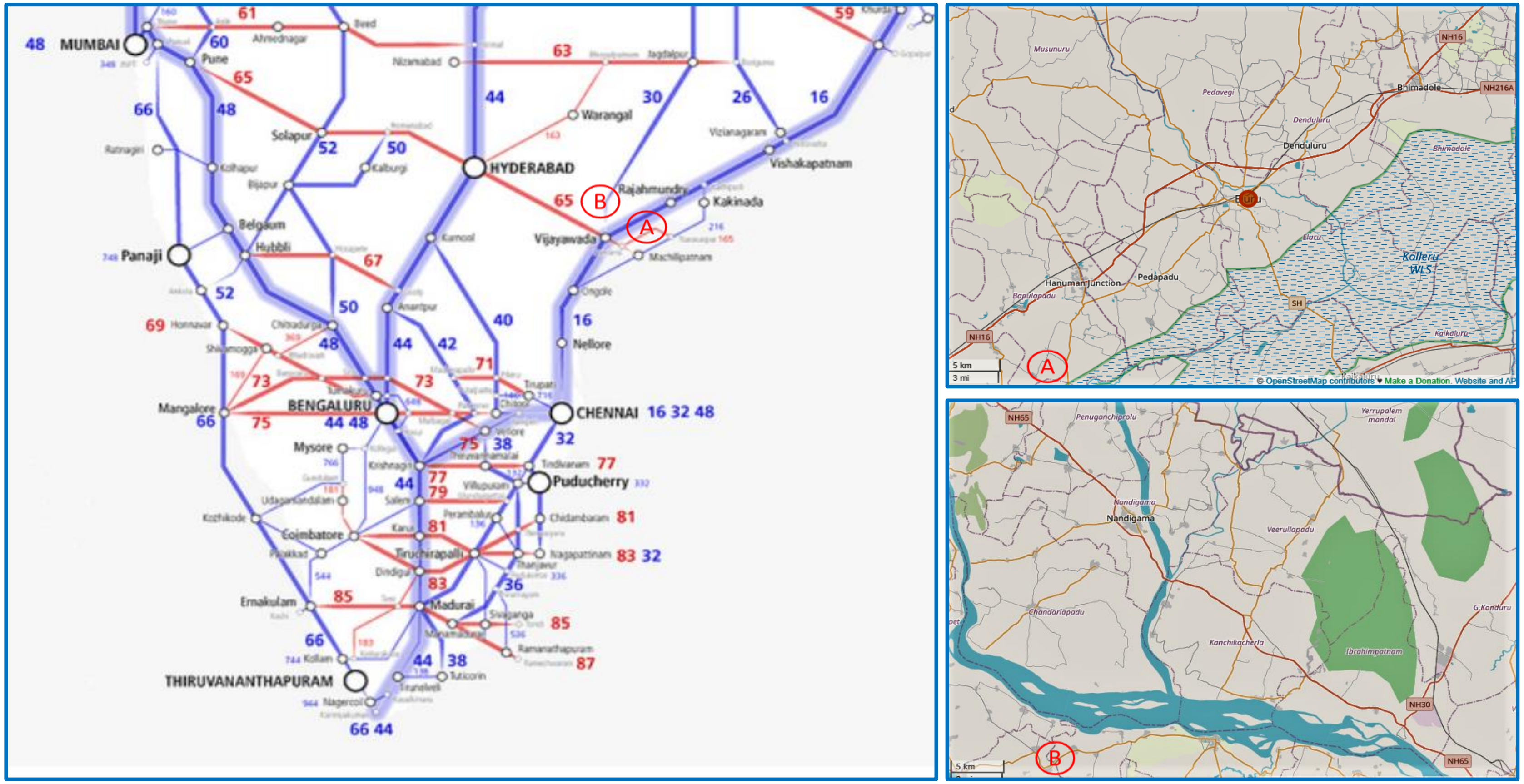


Figure 4.2 Map locations of the field (A) Gundugolanu Section - Eluru, A.P. (B) Nandigama Section, A.P.

(Source: https://en.wikipedia.org/)

It was well-established that the top 10 to 15 mm (on an average 12 mm) layer usually undergoes rapid aging process due to the influence of oxygen and UV rays present in the atmosphere while the bitumen in the bottom portion remains literally unaged (Kim et al. 2013). With this background, field cores were sliced in to 12 mm (top) and 35 ± 5 mm (this thickness varied slightly based on field compaction conditions) (bottom) across the thickness. The top and bottom slices were labelled accordingly and investigated separately. The schematic and the actual sample of sliced cores are presented in Figure 4.3.

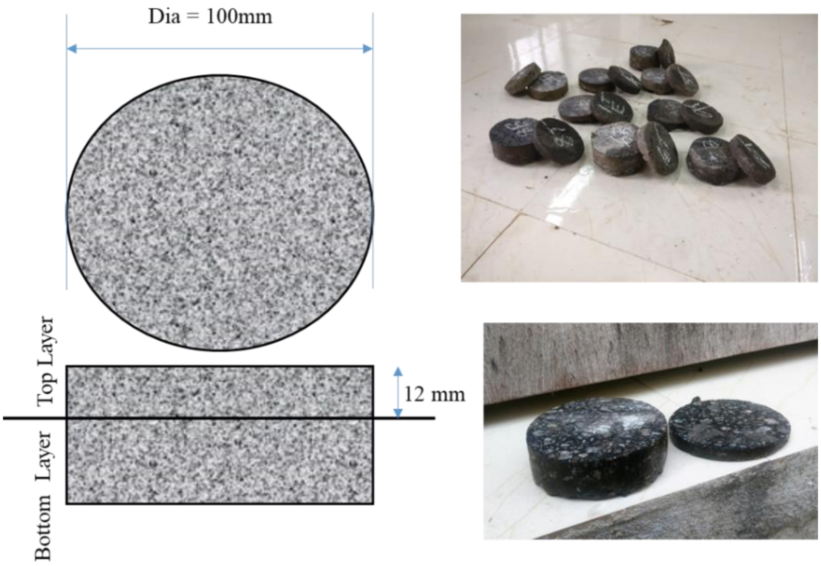


Figure 4.3 Schematic and actual photo of the sliced field core sample

Further, bitumen and aggregates were separated for the top and bottom slices of the numbered field cores using Soxhlet apparatus. The records collected from the site office revealed the adoption of Ministry of Road Transport and Highways (MoRT&H), Government of India's Bituminous Concrete Grade 1 (BC- Grade I) and VG 30 grade bitumen for both the highway sections.

4.3 Preparation of laboratory bituminous concrete specimens

The specimens for this study are cast as per Marshall's mixture design, detailed in MS 2 manual (Asphalt Institute 2014).

4.3.1 Details of Sasobit (Warms mix additive)

Sasobit, with the chemical formula of C_nH_{2n+2}, is a warm binder produced by Sasol Wax Company located in South Africa. It is generated by gasification of coal and contains a long chain of aliphatic hydrocarbons. Sasobit, which is a synthetic microcrystalline wax, has finer crystalline structure and longer chain length than the natural asphalt waxes. The properties of Sasobit used for the present study are summarized and presented in Table 4.3.

The main chain length of hydrocarbons present in Sasobit is of the range 40 – 115 atoms of carbon, while the natural bitumen paraffin waxes have only 22 – 45 atoms of carbon (Syroezhko et. al. 2011). These long range of carbon atoms present in Sasobit keeps the wax in a medium of solution, in turn decreasing the viscosity of the binder, improving the plastic limit and therefore, causing the increase in variation of melting temperatures of bitumen (Wasiuddin et. al. 2011).

Table 4.3: Properties of Sasobit used

Property	Value
Form:	Solid
Colour:	White
Odour:	Odourless
pH-value:	Not applicable.
Congealing point:	101 °C
Flash point:	>200 °C
Explosion Lower limits:	15 g/m3
Density at 20 °C:	0.9 g/cm3
Brookfield viscosity at 135°C	12 cP

The Sasobit has a melting point of about 100 ° C and it forms a homogeneous mixture when added with the bituminous binder at temperatures greater than 116 ° C. When temperature is increased beyond the melting point of Sasobit, the wax liquefies and reduces the viscosity of bitumen binder, consequently decreasing the bitumen mix production temperature by 20 to 30 ° C (Jamshidi et al. 2013). Sasobit, when added to bitumen binder, makes the binder act like a Newtonian fluid at elevated temperatures and non-Newtonian fluid at lower temperatures, below the Sasobit's melting point.

The main advantages of using Sasobit, as warm mix additive, can be listed (Hurley et. al. 2005):

- Less aging because of decreased mixing and construction temperatures.
- Sasobit lowers the air voids which indicates the reduction in the optimum binder content.
- Sasobit modified mixes have improved compactability and workability.
- Sasobit does not affect the resilient modulus of the bitumen mix, so it can be used without affecting the performance of the mix.
- The rutting potential is decreased for bitumen mixes when Sasobit is added.

Considering the success of Sasobit as a warm mix additive as observed by Hurley et al. (2005), Sasobit is considered for this research activity.

4.3.2 Hydrated lime as a filler

Hydrated lime is used as an additive in bitumen concrete mixtures for very long time. The use of hydrated lime had increased many folds from 1970s due to the need of better durable mixes. Hydrated lime is accepted as the most effective additive for bitumen mixes and all the construction agencies around the world are using hydrated lime as a regular additive. Hydrated lime is generated by hydrating the quicklime or calcium oxide CaO.

In India, MoRT&H specification (Specifications for Road and Bridge Works, 5th revision, published by the Indian Roads Congress on behalf of the Govt. of India, Ministry of Road Transport and Highways, 2014) recommends the use of hydrated lime up to 2%. In USA, AASHTO M 303 and ASTM C 1097 specifies the use of hydrated lime in bitumen concrete mixtures. In Europe, specifications for hydrated lime and quicklime are found in European standard for construction and civil engineering applications, EN 459-1.

Hydrated lime is generally considered as a mineral filler due to its origin and powder form. The comparison between hydrated lime and the mineral filler is presented in Table 4.4. The European standards for hot-mix bitumen concrete series EN 13108-1 to 13108-7, specifies cements and hydrated lime as fillers. As hydrated lime is treated as filler by these agencies, the hydrated lime can be estimated and characterized using the provisions laid for aggregates for its use in bituminous concrete mixtures as described in EN 13043.

Table 4.4: Comparison of mineral fillers and hydrated lime (Lesueur et al. 2013)

Property	Method	Unit	Hydrated lime	Mineral filler
Particle density	EN 1097-7 / AASHTO T84	Mg/m^3	2.2	2.6-2.9
Voids in dry compacted filler	EN 1097.4 - NAPA IS - 127	%	60-70	28-45
Bitumen number	EN 13179 - 2	-	70-120	40-50
Mass in kerosene	EN 1097 - 3	Mg/m^3	0.3	0.5-0.9
Specific surface area	BET (N_2 ADSORPTION)	cm^2 / g	150,000-200,000	14,000-95,000

The advantage of using hydrated lime as filler is primarily due to the improvement in the following (Lesueur et al. 2013):

- The resistance of chemical aging in bituminous mixtures.
- The resistance to the damage caused by moisture.
- The mechanical properties of the mixture namely strength, modulus, fatigue, thermal cracking and rutting resistance.

These improvements are explained by aggregate modification where the surface precipitation of calcium ions, flocculation of clay occurs and modification of binder due to the acid base neutralization (Sebaaly 2007).

Over the past fifty years, hydrated lime has become very important additive for bituminous mixtures, with its strongest presence throughout the world. Currently, it is not only considered as just a filler but also as an excellent additive that enhances the serviceability of bituminous mixtures. Hence, hydrated lime is selected for this study, with a specific target of investigating its anti-aging properties.

4.3.3 Tests performed on bitumen

As per the construction records collected at the respective site offices, it was found that VG 30 grade bitumen was being used. This aspect was reconfirmed by carrying out the fundamental tests on the extracted bitumen from the field cores, after duly considering the aging related changes that would have occurred.

Important fundamental tests on the VG30 grade bitumen were carried out before the aging related investigations. A minimum of three samples were tested to verify the repeatability and reproducibility of the results. The test results are summarised and presented, along with the standards being followed, in Table 4.5.

Table 4.5: Summary of virgin bitumen (VG – 30) test results

Property	Value	Threshold limits as per IS 73
Penetration as per IS 1203	50.44 dmm	> 45
Softening point as per IS 1205	54.8 ° C	> 47
Ductility as per IS 1208	80 cm	>40
Specific gravity as per IS 1202	1.005	---
Absolute viscosity at 60°C, Poises	3100	2 400-3 600
Tests on residue from RTFOT		
a. Ductility at 25°C, cm	44	40 min
b. Viscosity ratio at 60°C	3.6	4.0 Max

4.3.4 Tests performed on aggregates

As per the construction records collected at the respective site offices of both the National Highways from where the cores were extracted, it was found that grey coloured granite aggregates were being used at both the sites. Hence, similar aggregates were procured from a matching local quarry and used for further. As a prerequisite for the mix design and other investigations being planned, the aggregates were subjected to the following fundamental tests

- Shape Test (Combined Flakiness and Elongation Indices)
- Crushing Value Test
- Impact Test
- Los Angeles Abrasion Test
- Specific Gravity Test

Bulk quantity of aggregates was reduced to required quantity using riffler sample splitter. A minimum of three samples were tested to verify the repeatability and reproducibility of the results. The results of all the tests are summarised and presented, along with the standards being followed, in Table 4.6.

Table 4.6: Summary of results of tests done on aggregates

Aggregate Property	**Average of observed Values**	**Threshold limits as per MoRT&H**
Combined Flakiness and elongation Indices as per IS: 2386 Part 1	16.4 %	Max 30%
Crushing Value as per IS: 2386 Part 4	24 %	< 30%
Specific gravity as per IS: 2386 Part 3	2.624	2.5-2.9
Impact Value as per IS: 2386 Part 4	24 %	Max 27%
Abrasion Value as per IS: 2386 Part 4	28.9%	Max 35%

4.3.5 Aggregate gradation and job mix formula

Particle size distribution or gradation is one of the most influential aggregate characteristics, controlling the performance of bituminous concrete mixture under the combined influence of wheel and environmental loads.

Four different sizes of the aggregates (20 mm, 10 mm. 6 mm and fines including dust and sand sized particles) were procured for the experimentation. MoRT&H defined grade I Bituminous Concrete (BC-I), which was used in both the locations from where the core specimens were collected, was targeted, by judiciously blending the aggregates. Excel solver was employed to get the appropriate blend after duly finding the particle size distributions of each of the aggregate heaps defined by 20 mm, 10 mm, 6 mm and fines. Each of these aggregate heaps were named as Heap 1, Heap 2, Heap 3 and sand respectively. The particle size distribution of all the heaps is presented in Table 4.7. The same is shown in graphical form in Figure 4.4 for easy understanding.

Table 4.7: Sieve analysis of aggregates

IS Sieve Size (mm)	Aggregate Size (mm)			
	Heap 1	Heap 2	Heap 3	Sand
	Cumulative % Passing			
19.000	92.89	100.00	100.00	100.00
13.200	19.19	100.00	100.00	100.00
9.500	0.63	98.61	85.52	100.00
4.750	0.00	34.44	13.50	100.00
2.360	0.00	7.53	3.69	88.14
1.180	0.00	4.48	2.29	64.47
0.600	0.00	3.23	2.06	52.13
0.300	0.00	2.56	1.82	32.40
0.150	0.00	1.97	1.68	5.83
0.075	0.00	1.08	1.21	2.94

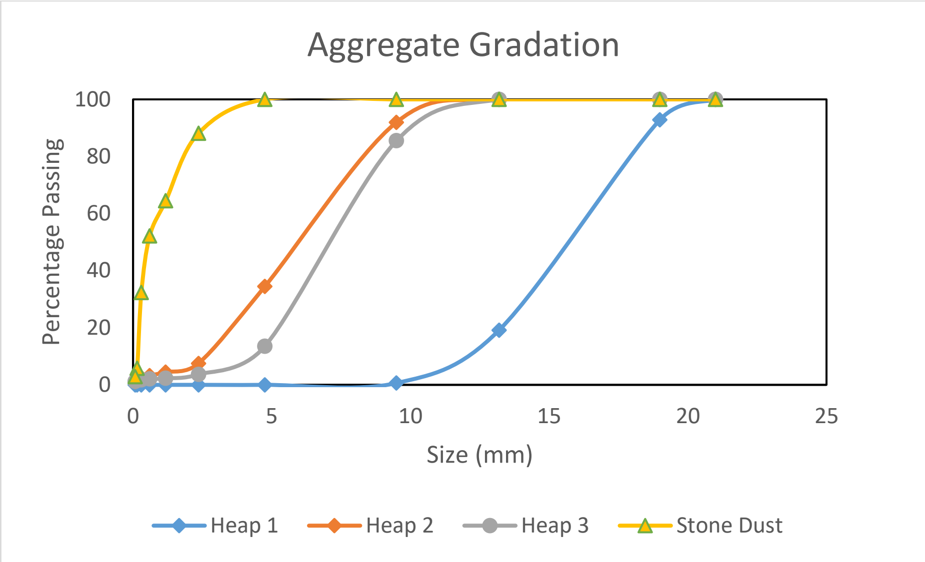


Figure 4.4: Aggregate Gradation

The Excel Solver was employed to find the percentage feed from each of the heaps to achieve the targeted mid-point gradation for MORT&H's BC I. The results of this exercise are summarised and presented through Table 4.8 and the same is pictorially depicted through Figure 4.5.

Table 4.8: Job Mix Formula

IS Sieve Size (mm)	MoRT&H Specification			% Feed					JMF
	Lower Limit	Upper Limit	Mid Limit	Heap 1 32	Heap 2 11	Heap 3 15	Sand 40	Lime 2	Percent Passing (%)
26.5	100	100	100	-	-	-	-	-	100
19	90	100	95.00	29.72	11.00	15.00	40.00	2	97.72
13.2	59	79	69.00	6.14	11.00	15.00	40.00	2	74.14
9.5	52	72	62.00	0.20	10.85	12.83	40.00	2	65.88
4.75	35	55	45.00	0.00	3.79	2.02	40.00	2	47.81
2.36	28	44	36.00	0.00	0.83	0.55	35.26	2	38.64
1.18	20	34	27.00	0.00	0.49	0.34	25.79	2	28.62
0.6	15	27	21.00	0.00	0.36	0.31	20.85	2	23.52
0.3	10	20	15.00	0.00	0.28	0.27	12.96	2	15.51
0.15	5	13	9.00	0.00	0.22	0.25	2.33	2	4.80
0.075	2	8	5.00	0.00	0.12	0.18	1.18	1.86	3.34

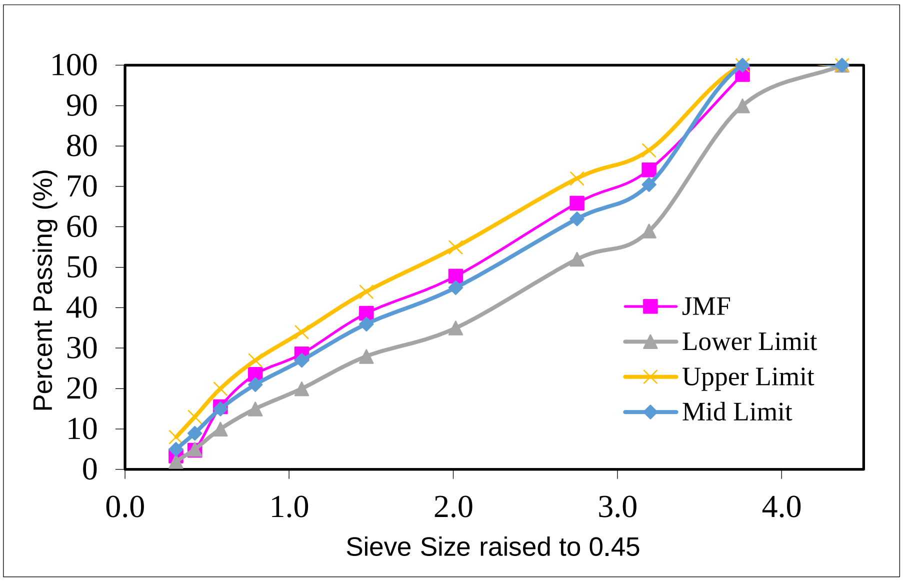


Figure 4.5: Design Aggregate Gradation

4.3.6 Optimum Binder Content (OBC)

Mix design reports, collected from the respective site offices of the locations from where the cores were extracted, revealed that the Marshall's mix design was employed. Hence, in this study also, the requisite specimens were cast after getting the optimum binder content through Marshall's mix design process. Following stages, as suggested in MS 2 manual, were being followed to arrive at optimum binder content.

- Selection of bituminous binder
- Selection of aggregates
- Sample preparation with standard conditions mentioned in Asphalt Institute's MS 2 manual
- Determination of stability and flow values using the standard machine, capable of applying the load at the loading rate of 50.8 mm / minute.
- Finding volumetric properties of the mix and ensuring that the mix is having better properties than the threshold limits mentioned in standards
- Finding the optimum binder content for final implementation

Following the standards provided in MS 2 manual, 100 mm (4”) cylindrical specimens were cast with aggregate blend obtained from the JMF (explained in 4.3.5). Bitumen contents of 4.5% to 6 % with 0.5 % incremental steps were tried. For each of the trial bitumen contents, 3 specimens were cast to ensure reproducibility of the test results. After extraction, the specimen height was measured at 4 locations along the circumference and the average height was computed. Also, the mass of the specimen in air as well as in water was found for further use. Prior to testing, these specimens were subjected to the hot water treatment for 30 minutes at 60 ^{0} C followed by stability and flow testing.

The average representative values of Marshall Stability, Marshall Flow, percentage Air Voids (av) and specific gravity for each set of specimens is summarized and presented in Table 4.9.

Table 4.9: Marshall’s mix design results

% Bitumen by weight of aggregate	**4.50**	**5.00**	**5.50**	**6.00**
% Bitumen by weight of the mixture	4.31	4.78	5.21	5.67
Marshall stability KN	7.31	10.95	8.16	7.22
Bulk Specific gravity of the specimen	2.29	2.37	2.36	2.28
% Air voids	6.46	4.30	2.82	2.22

The following requisite graphs were plotted from the results obtained from testing and the same are presented in Figure 4.6 (a to d).

- Percentage of bitumen and corrected Marshall stability of the mix.
- Percentage of bitumen and Marshall flow of the mix.
- Percentage of bitumen and Percentage of Air Voids in the total mix.
- Percentage of bitumen and Bulk specific gravity of the mix.

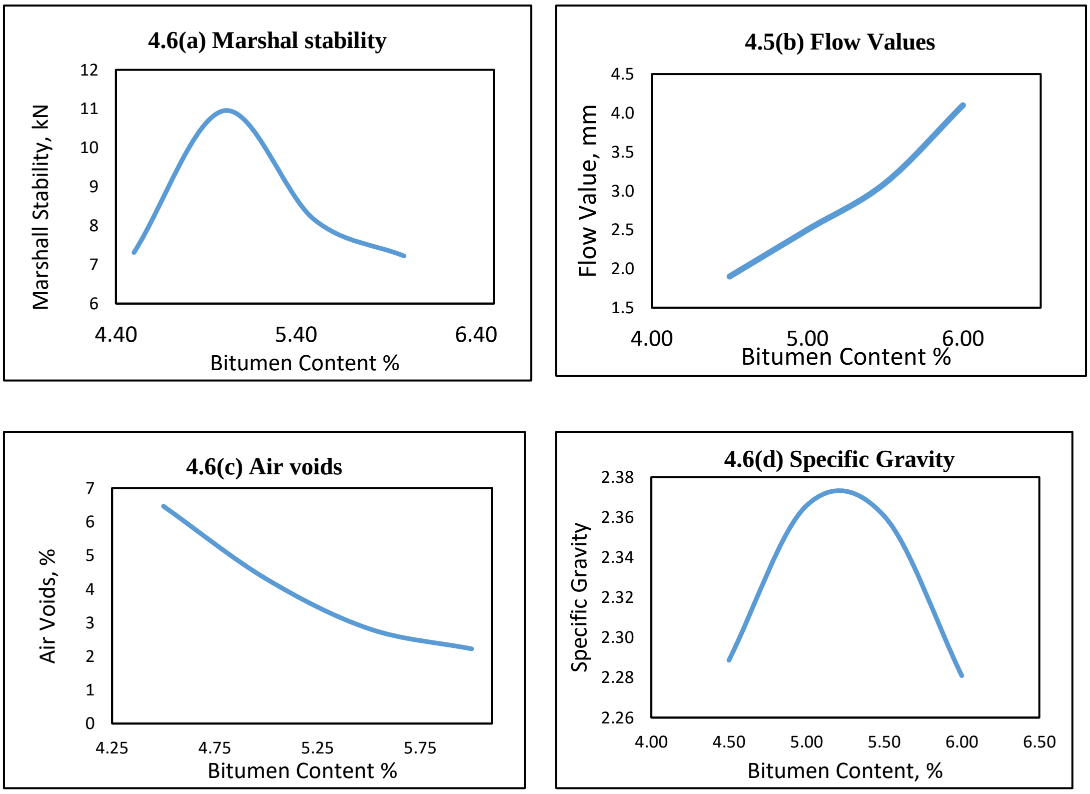


Figure 4.6: Marshall's graphical plots

From Figure 4.6 (c), the optimum binder content of 5.1% corresponding to 4 % air voids (as per MS-2 guidelines) was selected for the preparation of laboratory specimens.

4.3.7 Preparation of laboratory test specimens for artificial aging and testing

It was observed from the field records that neither hydrated lime nor any kind of warm mix additives were being used in the field core specimens. Hence, requisite number of specimens were cast without any of these additives. These specimens were named as Case I specimens. Oven exposure regimes were fixed as 0 days (control specimens), 1 day, 3 days, 5 days, 7 days, 10 days and 15 days (a total of 6 aging regimes plus one control specimen set, without aging). The aging regimes were decided based on the experience gained with a few specimens, which were studied during a pilot study carried out with day wise progression of aging of the specimens.

To optimize the quantity of Sasobit warm mix additive, Marshall's cylindrical specimens, with varying quantities of Sasobit along with a fixed 2% hydrated lime, used as an anti-stripping agent, were cast. The properties of the hydrated lime added were presented in Table 4.10.

Table 4.10 Chemical properties of hydrated lime

Property	Value
Appearance	White
Calcium Hydroxide $Ca(OH)_2$	80%
Moisture %	0.40%
Magnesium (MgO)	1.05%
Silicon Dioxide (SIO_2)	1.50%
Calcium Oxide (CaO)	60.50%
Calcium carbonate ($CaCO_3$)	4.50%
Iron Oxide (Fe_2O_3)	0.15%

For each of the combinations, 3 Marshall's specimens were cast. Marshall's stability and flow values were found after curing the specimens at 60 ^{0}C for 30 minutes. Average values of stability and flow are summarized and presented in Table 4.11.

Table 4.11 Marshall's stability and Flow values for bituminous concrete with hydrated lime and varying proportions of Sasobit

Percentage of Sasobit	Average Marshall's Stability (Kgs)	Average Flow Value (mm)
0.00	1080.1	3.8
0.50	1092.4	3.3
1.00	1101.7	3.1
1.50	1110.0	2.8
2.00	1159.5	1.8

From Table 4.11, it can be observed that the Marshall's stability has increased quite marginally while there was a noticeable reduction in the flow values. This aspect was observed to be logical as the introduction of Sasobit makes the mixtures brittle, especially at low temperatures such as 60 ^{0} C. Flow value is crucial, and it is warranted that this value shall be in the range of 2 mm to 4 mm. However, when 2 % Sasobit is added, the flow value has dropped below 2 mm, thus making the mixture unusable for application as per MS-2/MoRT&H. Hence, a Sasobit content of 1.5 % by the weight of bitumen is decided as the optimum content and is being used throughout the current research. Specimens cast with hydrated lime and Sasobit were labelled as Case II specimens and these were subjected to all the investigations to determine the efficacy of these additives in controlling the aging phenomenon. The mixture properties of the field specimens, laboratory prepared Case I and Case II samples are presented in Table 4.12.

Table 4.12 Mixture properties of field, Case I and Case II specimens

	Day 1 data from field office		**From extracted (aged) field core**		**Case I**	**Case II**
	NH 65	**NH 16**	**NH 65**	**NH 16**		
Binder content	5.1	5.1	5.1	5.1	5.1	5.1
Gmm	2.497	2.473	2.497	2.473	2.543	2.519
Gmb	2.302	2.29	2.405	2.379	2.37	2.34
Air Voids, %	7.8	7.4	3.6	3.8	6.8	7.1

For each of the chosen post-aging tests (Marshall's test, Indirect Tensile Strength: ITS and Tensile Strength Ratio: TSR), under all the aging regimes (0 days, 1 day, 3 days, 5 days, 7 days, 10 days and 15 days), for both the cases (Case I and Case II); 9 specimens each were cast (instead of usual 3 specimens) to ensure that the results are reproducible. A total of 378 test specimens were cast for aging investigations, following MS2 guidelines carefully.

4.4 Accelerated aging using modified AASHTO R30 oven

During the preparation of bituminous concrete mix, short term aging occurs since both aggregates and bitumen are subjected to heating at high temperatures. Once the mix is placed and compacted at the site, the long term aging process gets initiated. Hence, during the current study, bituminous concrete mix was subjected to short term aging before the compacted specimens were being subjected to long term aging process. AASHTO R30 recommended short term aging process was followed. Process involved in the short term aging process is detailed below for reference

4.4.1 Short Term Oven Aging (STOA) of bituminous concrete mix

The bituminous mixture was distributed in a flat pan to ensure a thickness in the range of 25 to 50 mm and was kept in an oven maintained at a temperature of 135 ± 3 °C for an un-interrupted duration of 4 hours. The mixture in the pan was stirred once in every 30 minutes to maintain uniform aging. This short term aged mixture is expected to exhibit similar characteristics of the mix ready to be placed in the field. This mix was then compacted with the Marshall's standard hammer. Further, these specimens were subjected to long term aging as detailed in the following sections.

4.4.2 Long Term Oven Aging (LTOA)

The long term aging protocol, provided by AASHTO R30, stipulates an exposure of the specimens at 85 ^{0} C for an un-interrupted period of 120 hours, to simulate the long term aging process. It was also suggested to keep the aged specimens inside the oven for an extended period of 16 hours, after stopping the heating process, to ensure that the specimens attain room temperature gradually.

A pilot study was carried out during the present study and results were presented in the Table 4.13. It was revealed that the aging achieved with 120 hours of oven exposure is nowhere near the aging observed in the field. Hence, it was decided to modify the AASHTO R30 process to suit the conditions prevalent at the locations from where the cores were collected. The details of the modifications done with the oven are detailed below.

Table 4.13 Physical properties of binder aged as per AASHTO R30 procedure

Aging	Penetration (dmm)		Softening Point (° C)		Ductility (cm)	
Days	**Case I**	**Case II**	**Case I**	**Case II**	**Case I**	**Case II**
0	50.4	50.4	54.8	54.1	80	80
1	45.2	48.1	55.4	54.1	72.5	75.5
3	42.8	46.6	56.1	55.6	67.5	73.5
5	41.2	45.3	57.1	56.7	66.5	72.5
	Penetration (dmm)		**Softening Point (° C)**		**Ductility (cm)**	
NH 65 Top	14.3		97.1		15.5	
NH 65 Bottom	16.0		70.5		20.5	
NH 16 Top	13.3		97.9		13.5	
NH 16 Bottom	15.3		71.1		18.5	

4.4.2.1 Oven modification for aging

An existing double walled electrical oven, of inner dimensions 90cm X 90cm X 90cm, heating range between 50 ° C to 400 ° C, fan speed of 1400 rpm, with proven repeatability standards, was chosen for the modifications and further use for long term aging process. The oven had a heavy-duty single door with hinges and strong locking mechanism, helping in minimizing the heat loss or escape of hot gases from the oven. It had four specious compartments where good number of specimens or material can be kept. A fan on the top portion of the oven was arranged for circulating the gases inside the oven and maintaining the temperature uniformly throughout the oven. Following modifications were done with this oven to suit the targeted long term aging process.

- Oxygen supply lines (0.25-inch copper tubes) were introduced, to expedite the aging process, from one of the sides of the oven by drilling a hole and sealing it tightly after the copper tube line was introduced.

- Pure Copper tube, with holes drilled with one inch spacing throughout the length, was selected for its ability to withstand high temperatures. Copper tube was run all over the surface of three walls of the oven (i.e., two side walls, one back wall) and on the bottom surface in multiple loops.
- In RTFOT test the air is supplied into the apparatus at a rate of 4000 ml/min. As this air is supplied for 8 samples through 15 rotations, rate of flow of the air is around 2000 ml/hr. In the modified oven, as the pure oxygen is supplied, instead of air, for the present study we have finalized the oxygen flow as 1000 ml/hr. to achieve similar level of aging in the modified oven.
- Flow of oxygen was adjusted to see that the pressure remained very close to 1 atm inside the oven throughout the aging process and flow of oxygen was controlled using pressure regulator and flow is measured using flow meter as shown in Figure 4.7.

The modified oven, with interiors and oxygen supply mechanism, is shown in Figure 4.7.

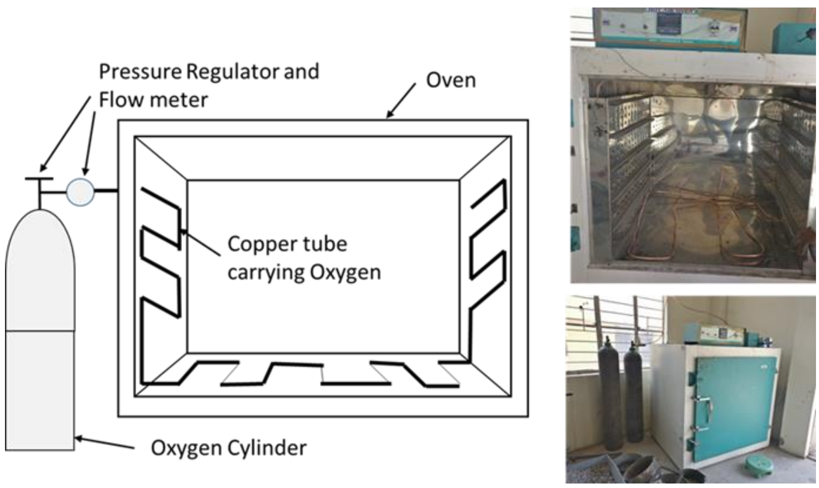


Figure 4.7: Schematic diagram and actual photo of modified oven

4.4.2.2 Long term aging of specimens using modified AASHTO R30 process

Bituminous concrete specimens, cast with short term aged mix, were subjected to long term aging process under the following exposure conditions

- Unmodified bituminous concrete specimen with exposures of 1 day, 3 days, 5 days, 7 days, 10 days and 15 days at 85 ^{0} C after short term aging of the un-compacted mixture at 135 ^{0} C. These are called Case I samples with 1-day aging are named CID1, with 3-day aging as CID3 and so on.
- Sasobit and hydrated lime added bituminous concrete specimen with exposures of 1 day, 3 days, 5 days, 7 days, 10 days and 15 days at 85 ^{0} C after short term aging of the un-compacted mixture at 135 ^{0} C. These are called Case II samples with 1-day aging are named CIID1, with 3 day aging as CIID3 and so on.
- Repeatability and reproducibility were ensured by taking a minimum of 9 specimens for each of the trials.
- After the completion of aging, the specimens are brought back to the room temperature by powering off the oven and keeping the samples inside the oven for 16 hours to bring the temperature back to room temperature.

The specimens, that are exposed to long term oven aging and brought back to room temperature, were subjected to volumetric measurements, stability, flow values, In-direct tensile strength (ITS), Tensile Strength Ratio (TSR) as detailed below.

4.5 Testing of laboratory aged bituminous concrete specimens

The specimens (of both Case I and II), aged for 0 days, 1 day, 3 days, 5 days, 7 days, 10 days and 15 days, were subjected to the standard laboratory investigations to investigate the following properties:

- Marshall Stability and flow test - ASTM D5581
- Indirect Tensile Strength (ITS) - As per ASTM D 6931
- Tensile Strength Ratio (TSR) - As per ASTM D4867 / D4867M

4.6 Bitumen recovery from laboratory aged and field specimens

Bitumen recovery from laboratory aged and field core specimens, was one of the key stages during the present research activity. Bitumen was separated from the differently aged and tested laboratory specimens (discussed in section 4.5) using reflux method (Soxhlet extractor). In addition, the bitumen extraction is being carried out from the field cores also for further investigations. Important details of field cores are presented below for reference.

To determine the level of aging that occurs in the field, bituminous concrete cores of the in-service pavements were collected from National Highways with the permission from the National Highway Authority of India. The cores were collected from NH 16, with 7 years of service life and NH 65 with 5 years of service life. The variability in the performance of in-service pavements depend on traffic level, variations in structural design, quality of materials used, weather conditions, mix design and maintenance activities that were taken up during the service life etc. With a view to reduce this variability, the sample cores were collected from the sections of the road that were laid during approximately the same time. A homogeneous and representative section of one kilometer was selected on both the highways for the collection of cores. Within the selected stretch, three different equi-spaced locations were chosen to be representing the whole kilometer. Again, from each of these three locations chosen, three core specimens were collected to check the repeatability and reproducibility. Nine (9) cores each from NH 16 and NH 65 were collected with standard coring practices.

It is a well-established fact that the top 10 mm to 15 mm layer of pavement section usually undergoes rapid aging process due to the influence of oxygen and UV rays present in the atmosphere while the bitumen in the bottom portion remains unaged. It is with this premise that the field cores were sliced in to 12 mm (top) and 51.5 mm (this thickness varied slightly based on compacting conditions) (bottom) across the thickness. The top and bottom slices were labeled accordingly and investigated separately. Details of sliced cores are presented in Figure 4.8 for reference.

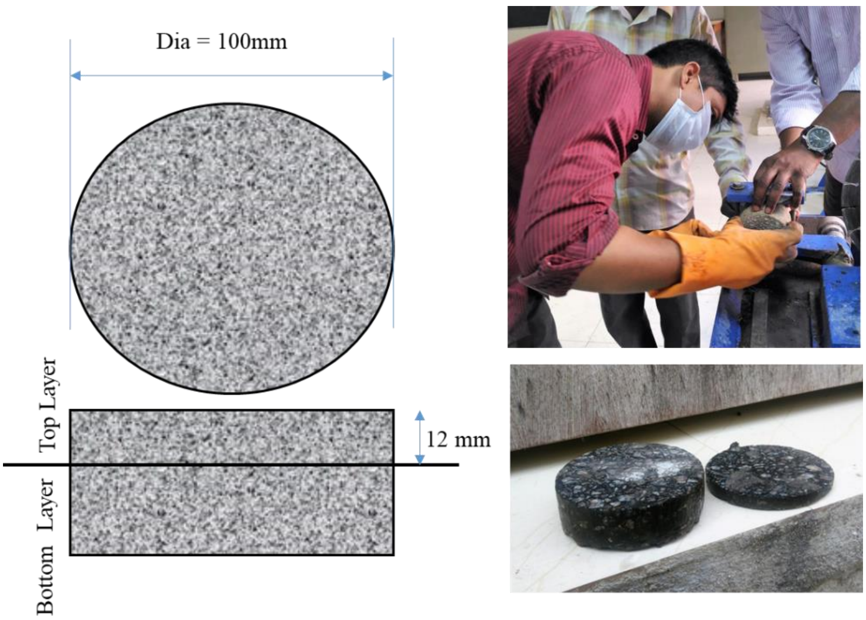


Figure 4.8: Top layer slicing of field cores

According to ASTM D 2172, there are different methods of bitumen extraction from mixtures. These are:

- Centrifuge Method
- Vacuum Method and
- Reflux Method

Among these methods, reflux method is the most popular one due to its availability and ease of use. For this study, Soxhlet extractor, shown in Figure 4.9, with a percolator (boiler and reflux) which circulates the solvent, a thimble (with a thick filter paper, to retain the solid to be extracted, a siphon mechanism, to empty the thimble periodically and a reflux condenser at the top of the extractor, was being used to separate bitumen from the bituminous concrete samples. The solvent used for extraction of bitumen was Toluene. The main precaution that had been taken while using this method was that the bituminous material is presoaked in the solvent before commencing the extraction to reduce the extraction time substantially. This, in turn, helped in keeping the bituminous mixture at lower temperatures, thus preventing uncontrolled aging.

Figure 4.9: Soxhlet extractor and extracted bitumen samples

The extracted / separated bitumen, after recovery from different specimens was kept in airtight steel containers with lids to avoid any further exposure and related oxidative aging. Required quantity of the extracted bitumen was taken from the steel containers, as and when needed, to carry out the tests on bitumen

4.7 Testing of aged bitumen

The main aim of this research, as mentioned already, is to investigate the influence of simulated and real time aging on the performance characteristics of bitumen and to compare the natural field aging with that of accelerated laboratory aging process. To fulfill this, the mechanical and microstructural investigations have been carried out on bitumen samples which were extracted, both from the laboratory aged and field aged specimens using Soxhlet extractor followed by simple distillation process for separating the solvent from the bitumen. This bitumen is further subjected to basic as well as advanced characterizations as listed below

- Penetration using Universal penetrometer (IS:1203-1978)
- Softening point using ring and ball apparatus (IS:1205-1978)
- Ductility test (IS:1208-1978)
- Viscosity by Brookfield's Rotational Viscometer (ASTM D4402 / D4402M-15)
- Rheological properties using Dynamic Shear Rheometer (DSR)

- Microstructural investigations of bitumen using Fourier-transformed infrared spectroscopy (FTIR) analysis, SARA analysis, field emission scanning electron microscope (FE SEM) analysis, energy dispersive X-Ray analyzer (EDX)

The first three tests listed above, namely penetration, softening point and ductility, are very common and hence description was not covered in this report. For the rest of the advanced tests, namely Viscosity using rotational viscometer, rheological properties with DSR and micro structural investigations; a brief description is given in the following sections for quick reference.

4.7.1 Viscosity using Brookfield's rotational viscometer (ASTM D4402 / D4402M-15)

The viscosity of the bituminous binder was found by Brookfield's rotational viscometer, shown in Figure 4.10. It is very common to find the viscosity of bitumen binders at high temperature range, observed during the manufacturing and construction stages. The viscosity of bitumen at a particular temperature of 135 °C is important because, the viscosity at this temperature is expected to control the ability of bitumen to be pumped, mix properly with aggregates and ensure proper workability.

The torque required for rotating an object in a fluid at a certain speed is dependent on the viscosity of that fluid. Primarily, rotational viscometers work with this principle. Rotational viscometer consists of a thermostatically controlled chamber, which can hold a sample of hot bitumen. A standard spindle, with the standard shape and total surface area, is lowered into the bitumen and rotated. The amount of torque required for rotating the spindle at a particular RPM is measured and is converted into the viscosity value of the bitumen.

Figure 4.10: Brookfield Rotational Viscometer

4.7.2 Dynamic Shear Rheometer

The dynamic shear rheometer (DSR) is an apparatus, shown in Figure 4.11, is used to perform the visco elastic analysis of bitumen. In DSR tests the sinusoidal strain is administered to the specimen, and stress resulted is noted as frequency function. As the strain is controlled, this test is called the 'strain-controlled testing'. This type of testing is more commonly used for visco elastic material like bitumen.

If any material is subjected to strain or stress profile as function of time, and if the strain is very small, then that will behave in linear visco elastic manner, which means there exists a linear relationship for stress and strain as defined by Hooke's law.

The procedure for testing the bitumen in DSR involves in applying the sinusoidal strains on bitumen samples sandwiched between rheometer's parallel plates, as oscillatory shear. The resulting stress amplitude is estimated by finding the transmitted torque through the specimen sample as a reciprocation to the applied strain. The strains that are induced during DSR testing must be kept small to ensure that the test is performed in the linear visco elastic region.

Figure 4.11: Dynamic Shear Rheometer

The frequency at loading "ω" is known as angular frequency. Sinusoidal stress is developed in response to the applied strain, this repose of the material is out of phase compared with the applied strain, which is called phase angle, "δ". The phase angle is specified as the difference between the stress and strain and called as phase lag. The phase angle for purely elastic materials is zero and is 90° for purely viscous materials, which makes this an important property in characterizing and understanding the Visco elastic behavior of bitumen. The ratio of resulted stress to the strain applied is known as complex shear modulus, G*, or complex modulus or simply stiffness. Relationship between the complex shear modulus (G*) in both the elastic portion (G′) and viscous portion (G″), and the phase angle (δ) is presented through Figure 4.12.

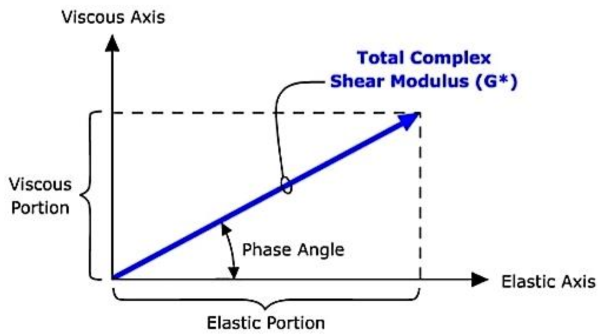


Figure 4.12: Relationship between the complex shear modulus (G*), the elastic portion (G′) and viscous portion (G″), and the phase angle (δ)

The in-phase component of complex modulus is known as the shear storage modulus, G' or simply storage modulus. The storage modulus represents the quantity of energy that is stored and let out elastically in every oscillation, and so it is also called as the elastic modulus. The storage modulus is given as Eq. 4.1:

$G' = G^* \cos \delta$ --------------- Eq. 4.1

The shear loss modulus or loss modulus G", is out of phase constituent of complex modulus which is given by Eq. 4.2:

$G'' = G^* \sin \delta$ ---------------- Eq. 4.2

The loss modulus defines the average energy dispersion rate present in the steady continuous oscillation seen in the dynamic shear test. The loss modulus is also called as the viscous component of complex modulus or viscous modulus.

The loss tangent is described as the ratio of the viscous component and elastic components of the complex modulus, or simply as the tangent of the phase angle given in Eq. 4.3.

$\tan \delta = \frac{G''}{G'}$ ------------- Eq. 4.3

4.7.3 Microstructural analysis of bitumen

Bitumen is heterogeneous with source specific variations in chemical constituents and composition. This becomes more complicated when some additives are added. As mentioned earlier, bitumen is principally made up of different hydrocarbons, which are complex in nature. Investigations carried out at micro / nano / molecular scales will certainly help in understanding this heterogeneity. Micro structural investigations help to understand the heterogeneity closely and accurately.

Also, it has been found in many studies that microscopic behavior of bitumen clearly indicates the way the bitumen is going to perform in macroscopic scale also (Allen et al, 2012, Poulikakos et al. 2014, Eberhardsteiner et al. 2015 and Li et al. 2020). Many techniques, under the category of micro structural investigations, have evolved over a period. However, the following techniques have been identified as the most relevant to suit the current study.

- Fourier-transform infrared spectroscopy (FTIR) Analysis
- Field Emission Scanning Electron Microscope (FE SEM) Analysis
- Energy Dispersive X-Ray Analyzer (EDX)

In addition to the above micro structural investigations, SARA (Saturates, Aromatics, Resins and Asphaltenes) analysis was also performed to have a comprehensive understanding of the changes in chemistry of bitumen with aging. A brief description of these advanced techniques is presented in the following sections before presenting the results.

4.7.3.1 Fourier Transformed Infrared Spectroscopy (FT-IR) Analysis

Infrared spectroscopy is one of the most important and accurate methods to characterize the chemical compounds of material by principle of absorption of infrared radiation of chemical bonds. The infrared spectrum is part of electromagnetic spectrum, which lies between microwave and visible region. Infrared spectroscopy can be performed on any type of material in any state.

The principle behind the Infrared spectroscopy is that, the infrared beam is transmitted into the material thereby activating the molecular bonds to agitate and rotate at distinct frequencies, reducing the energy at the specific frequency. These absorbed frequencies are identified with a detector. The notation Fourier transform infrared spectroscopy had originated from the mathematical process known as Fourier transformation, which is used to convert the complicated raw data to the actual useful spectrum.

As bitumen binder is a very complex material when Infrared spectroscopy is carried, we cannot see a very well-defined peaks, so instead of peaks, a range of wave numbers are used for evaluation of aging components. It is known that the oxidation causes aging of bituminous binders, therefore the presence and characterization of oxygen containing functional groups are important for detecting aging by means of FT-IR analysis. The essential information about oxidation groups like ketones and sulfoxides is given by

1800 – 600 cm^{-1} regions of spectrum. So, these functional groups, ketones (wave number 1700 cm^{-1}) and sulfoxides (wave number 1030 cm^{-1}), can be used as aging indicators.

The specific functional groups are identified according to their absorption spectrum in each region. The region between 1800 and 600 cm^{-1} is most important region for the study of ketones and sulfoxides as they represent the development of aging in bitumen.

For FT-IR testing, bitumen samples can be dissolved in toluene or any other standard bitumen solvent. During this research, toluene was used. The bitumen dissolved solution is then drop casted on the Potassium Bromide (KBr) plate to form a thin film as shown in the Figure 4.13. This KBr plate is then placed inside the FT-IR spectrometer for further analysis. The scanning over the sample is done in the mid infrared region of 600– 4000 cm^{-1}.

Figure 4.13: FT-IR Setup, sample preparation and samples

Care was taken to run the background scan on the KBr plate before running the test on the bitumen film to avoid the effects of any contamination. This FT-IR spectrometry was carried out at the room temperature and the resulted spectrum is analyzed for the absorption by the functional groups.

4.7.3.2 SARA Analysis

The characterization of the bitumen is usually done by saturates, aromatics, resins and asphaltene (SARA) fractions. The saturates, aromatics and resins falls under maltene fraction of bitumen. These maltenes and asphaltenes can be separated using n-Heptane as solvent, as maltenes are soluble and asphaltenes are insoluble in n-Heptane. Asphaltenes usually range in between five to twenty five percent by weight of bitumen. The relative amount of these asphaltenes and maltenes considerably affect the mechanical and physical properties of bitumen.

To separate asphaltenes and maltenes present in the bitumen, the conventional method, also known as Corbett separation scheme, was used during the present research. 100 ml of n–Heptane was added to each gram of bitumen taken (usually 10 to 15 grams) in Erlenmeyer flask. The sample was stirred while adding the n–Heptane and the solution was kept overnight. Later the mixture was filtered with Whatman grade – 1 filter paper through vacuum filtration. As the asphaltenes are insoluble in n–Heptane, they are retained on the filter paper. The retained solids are dried, weighed and reported as asphaltene fraction. From the passing solution, n–Heptane is separated by simple distillation and residue is collected, weighed and reported as maltene fraction. Important stages of the separation process are shown in Figure 4.14.

Figure 4.14. Corbett separation of asphaltene and maltene

4.7.3.3 Field Emission Scanning Electron Microscope (FE SEM) Analysis

The scanning electronic microscope employs a concentrated beam of high energy electrons to create different types of signals on the surface of solid-state specimens. These signals that originates from the electron sample interactions will disclose information about the specimen like the chemical composition, orientation of material making up the specimen, crystalline structure and external morphology or texture.

In many cases the data is taken from a selected area of outer surface of specimen and a two-dimensional image is produced that shows spatial variations of the features. The areas in-between 5 microns to 1 cm can be seen by scanning mode and employing conventional FE SEM techniques. Magnifications varying from 20 to 30,000X with spatial resolution of about 50 to 100 nm can be imaged.

To semi-quantitatively and quantitatively determine the chemical compositions, crystal orientations and crystalline structure, a selected point location analysis on the specimen can performed by FE SEM. The FE SEM machine is shown in Figure 4.15.

Figure 4.15: Field Emission Scanning Electron Microscope and samples

4.7.3.4 Energy Dispersive X-Ray Analyzer (EDX)

As observed in the previous section, the FE SEM uses electron beams to gather the information of the specimen at nanoscale levels. Apart from the main signals that produce grayscale image of the sample, there are many other signals which are a product of electron matter interaction and one of those signals is Energy dispersive X-ray (EDX).

The electron beam hits the specimen and causes the atoms of the sample to jump to higher energy shell leaving behind a hole. These holes are positively charged and will attract negatively charged electrons from other shells. When the electron is transferred from one shell to other shell, the energy difference of this transition is released in the form of an X-ray.

The released X-ray has energy which is related to the energy difference of the two shells involved. It depends on atomic number, which is unique property for every element. So, these released X-rays are fingerprints of each element present and can be used to identify the elements in the sample. The data generated by EDX is in the form of spectra, with peaks corresponding to the elements that are clearly present in the specimen.

The results along with analysis, for the following tests, is presented in the next chapter

- Penetration, softening point and ductility tests
- Viscosity using Brookfield's Rotational Viscometer
- Rheological properties using Dynamic Shear Rheometer
- Microstructural investigations of bitumen using Fourier-transformed infrared spectroscopy (FTIR) analysis, SARA analysis, field emission scanning electron microscope (FE SEM) analysis, energy dispersive X-Ray analyzer (EDX)

4.8 Summary

This chapter dealt with the description of various tests being conducted to achieve the objectives set. All the laboratory investigations, which were carried out, are shown through a flowchart, presented in Figure 4.16 for easy understanding.

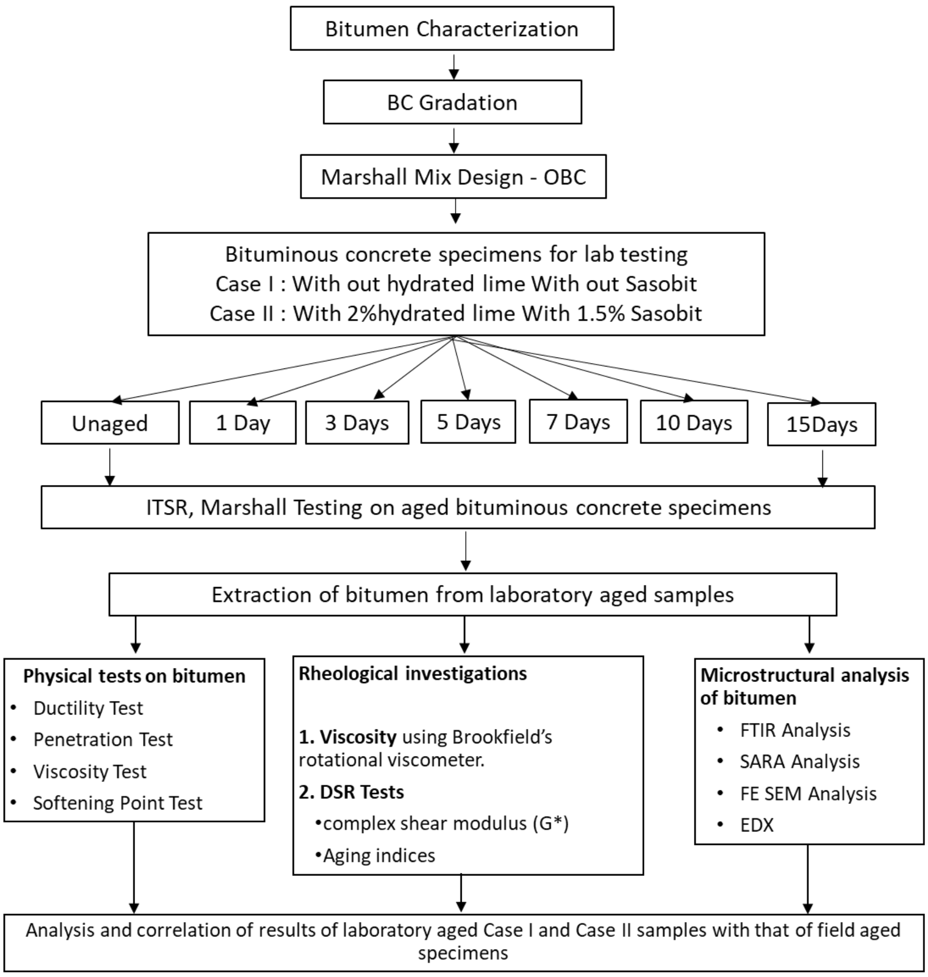


Figure 4.16: Summary of Laboratory Testing

CHAPTER 5

TEST RESULTS AND DISCUSSIONS

5.1 Introduction

In this chapter, the results of all the investigations along with the analysis and discussions, carried out during the present research, are presented.

Differently aged bituminous concrete samples along with the samples, not exposed to any aging (reference samples), were tested to find out the performance in terms of stability, flow, densities, indirect tensile strength (ITS) and tensile strength ratio (TSR). Reference codes, listed below, were followed while conducting these tests during the current research.

- Marshall Stability and flow test - ASTM D5581 and MS 2 Manual
- Indirect Tensile Strength (ITS) - As per ASTM D 6931
- Tensile Strength Ratio (TSR) - As per ASTM D4867 / D4867M

In addition, extracted bitumen samples from laboratory aged and field aged specimens were subjected to basic investigations like penetration, softening point, ductility and viscosity to verify the changes happening due to aging phenomenon. Further, micro structural investigations were also carried out to understand the changes in morphology, crystallinity and chemistry in the aged binder. The following tests were conducted under this category

- Viscosity by Brookfield's Rotational Viscometer.
- Rheological investigations (G *, G* Sin δ, G* / Sin δ and Multiple Stress Creep Recovery) using Dynamic Shear Rheometer (DSR).
- Fourier Transform Infrared Spectroscopy (FT-IR) to investigate the chemical reaction activity with aging.

- Saturates Aromatics Resins and Asphaltenes (SARA) analysis for understanding the change in asphaltene and maltene contents with aging.
- Field Emission Scanning Electron Microscopy (FE SEM) for investigating the surface morphology.
- Energy Dispersive X-ray analysis (EDX) for finding the elemental composition.

5.2 Results and investigations on laboratory aged bituminous concrete

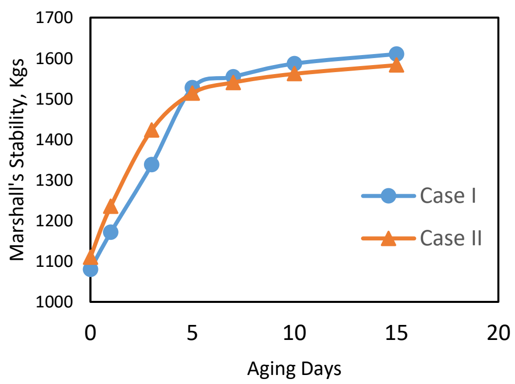
A summary of results of tests conducted on Case I and Case II specimens is presented below. It may be recalled here that, Case I refers to those specimens without any additives while Case II refers to those specimens with optimized quantities of hydrated lime and warm mix additive, Sasobit.

5.2.1 Marshall's stability and flow values for unaged and laboratory aged specimens

The results of Marshall's stability and flow values for Case I and Case II specimens are summarized and presented in Table 5.1 and graphically shown in Figure 5.1. It can be clearly seen that the advanced aging has induced incremental stability with corresponding reduction in flow values. This phenomenon can be attributed to the fact that the aging induces additional stiffness with compromised elastic responses. However, there was only a marginal difference in the stability and flow values between Case I and Case II specimens.

Table 5.1 Marshall's stability and flow value results with aging for lab aged specimens

Aging (Exposure in Modified Oven)	Marshall's Stability (Kgs)		Flow Value (mm)	
Days	Case I	Case II	Case I	Case II
0 (No aging)	1080.1	1110.0	2.5	2.9
1	1171.2	1235.6	2.3	2.8
3	1337.6	1423.1	2.1	2.5
5	1527.0	1513.9	1.8	2.1
7	1554.3	1540.2	1.5	1.9
10	1586.7	1561.6	1.3	1.7
15	1610.1	1582.9	1.1	1.4



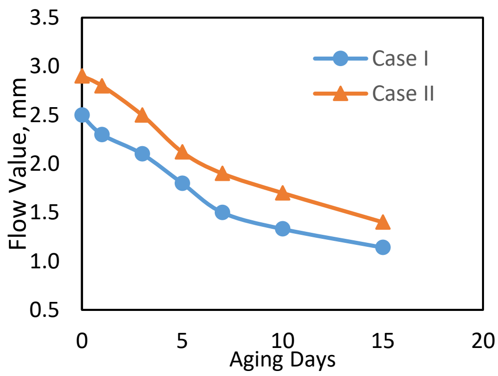


Figure 5.1. Variation in Marshall's stability and flow values of Case I and Case II specimens

With a view to get a deeper insight of the results, changes observed in the stability values of the progressively aged Case I and Case II specimens were worked out in terms of percentage change per day and presented in Table 5.2. It can be clearly seen that, during the initial stages of oven aging process, the change in Marshall's stability values were relatively higher (8.44% and 11.32%) for both Case I and Case II specimens respectively. With the advanced aging exposures, the corresponding changes have become lesser indicating that the rate of stiffening is falling with reduced influence of the aging at advanced exposure periods. It is interesting to note that the Case II specimens have resulted in accelerated stiffening than the Case I specimens during the initial aging periods. However, at advanced aging periods, the Case I specimens stiffened more dynamically than Case II specimens.

Table 5.2 Percentage increase in Marshall's stability

Aging period	Percentage increase of Marshall's stability in the aging periods	
	Case I	Case II
0-1	8.44	11.32
1-3	7.10	7.59
3-5	7.08	3.19
5-7	0.89	0.87
7-10	0.70	0.46
10-15	0.30	0.27

5.2.2 Indirect tensile strength (ITS) and tensile strength ratio (TSR) of unaged and laboratory aged specimens

Indirect tensile strength test for both 24-hour conditioned (at 60 ^{0}C) and unconditioned specimens (Case I and Case II) was carried out. In addition, tensile strength ratio (TSR) values were computed to assess the relative moisture sensitivity of the specimens after aging. The test results of ITS and TSR are summarized and presented in Table 5.3. For better clarity, TSR values are also presented graphically through Figure 5.2.

Table 5.3 Indirect tensile test results with aging of artificially aged specimens

Aging	ITS dry (MPa)		ITS- conditioned (MPa)		TSR	
Days	Case I	Case II	Case I	Case II	Case I	Case II
0	0.61	0.67	0.47	0.55	0.77	0.82
1	0.65	0.74	0.52	0.62	0.79	0.84
3	0.71	0.80	0.59	0.69	0.83	0.86
5	0.79	0.86	0.67	0.76	0.84	0.88
7	0.85	0.90	0.73	0.81	0.86	0.90
10	0.88	0.91	0.77	0.83	0.88	0.91
15	0.91	0.93	0.82	0.86	0.90	0.92

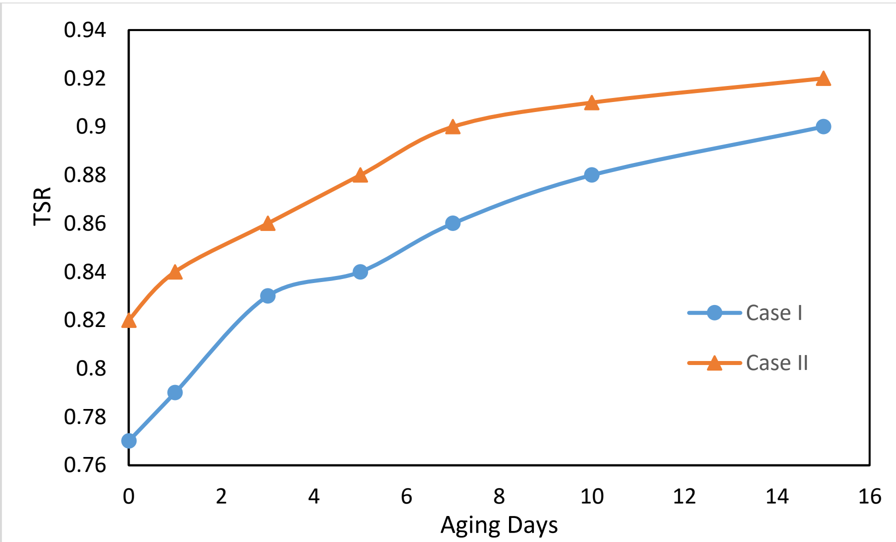


Figure 5.2. Variation in tensile strength ratio of Case I and Case II specimens

It can be observed from Table 5.3 and Figure 5.2 that the Case II specimens have outperformed the Case I specimens with increasing ITS (for both conditioned and un-conditioned cases) and TSR values, with progressing aging. This phenomenon can be attributed to the increased stiffness with aging process. Also, it can be noted that the difference of conditioned ITS for Case II specimens is more prominent than for Case I specimens, indicating the improved moisture sensitivity of the Case II specimens due to the addition of hydrated lime. Hydrated lime, being a very good anti-stripping agent, would have made the difference in this phenomenon.

The rate of change of ITS values, kept reducing for both conditioned and unconditioned samples, with increase in aging time. This was clearly observed from Table 5.4, where the diurnal variations in ITS and TSR values were computed and presented for different periods of aging. This phenomenon can be attributed to the increased stiffening of bitumen initially than when exposed to prolonged aging process. Loss of maltenes during the initial phase of aging is usually more pronounced and hence the above trend is justified. Also, the changes in TSR variation with time is better in Case II specimens due to hydrated lime.

Table 5.4 Percentage change per day in indirect tensile strength test for laboratory specimens

Percentage change (%) in ITS and TSR results (per day of oven exposure)						
	ITS Dry		ITS- conditioned		TSR	
Aging period	Case I	Case II	Case I	Case II	Case I	Case II
0-1	6.45	9.79	9.75	12.81	3.10	2.75
1-3	4.83	4.64	7.54	5.89	2.48	1.14
3-5	5.62	3.71	6.07	4.54	0.41	0.78
5-7	3.92	2.23	4.92	3.70	0.93	1.41
7-10	1.08	0.12	1.90	0.55	0.79	0.43
10-15	0.61	0.54	1.25	0.78	0.63	0.24

5.3 Results of investigations on extracted bitumen from laboratory and field aged specimens

5.3.1 Fundamental properties of extracted binder

It is a well-known fact that the basic properties of bitumen namely penetration, softening point, ductility and viscosity vary with changing SARA fractions in bitumen and these fractions keep changing with aging process. It is with this premise that the extracted binders are subjected to the following tests to understand the effect of aging process on bitumen. Standards followed for these tests are listed below for reference.

- Penetration using penetrometer as per IS:1203-1978 (ASTM D5)
- Softening point using ring and ball apparatus as per IS:1205-1978 (ASTM D36)
- Ductility test as per IS:1208-1978 (ASTM D113)
- Viscosity using Brookfield's rotational viscometer (ASTM D4402 / D4402M-15)

The results obtained by testing the binder extracted from laboratory aged samples are presented in Table 5.5 and the same are graphically shown in Figure 5.3. As per the standard trends, with increased aging exposure, the penetration and ductility values were observed to be dropping while the softening point values have increased. It can also be seen that the changes are more prominent for Case I unmodified samples when compared with modified samples (Case II), thus proving the efficacy of the modifiers in delaying the aging phenomenon. Interestingly, the changes were observed to be more prominent during the initial exposure.

Table 5.5 Physical properties of binders extracted from Case I and Case II samples

Aging	Penetration (dmm)		Softening Point (° C)		Ductility (cm)	
Days	Case I	Case II	Case I	Case II	Case I	Case II
0	50.4	50.4	54.8	54.1	80.0	80.0
1	37.0	40.0	57.7	56.6	61.5	68.5
3	32.2	34.5	63.5	58.7	58.5	64.5
5	28.2	29.2	65.1	61.5	54.0	57.5
7	24.7	25.5	67.8	64.7	51.5	54.5
10	20.0	21.2	75.7	72.2	33.5	40.5
15	13.5	14.0	87.4	86.2	12.0	16.5

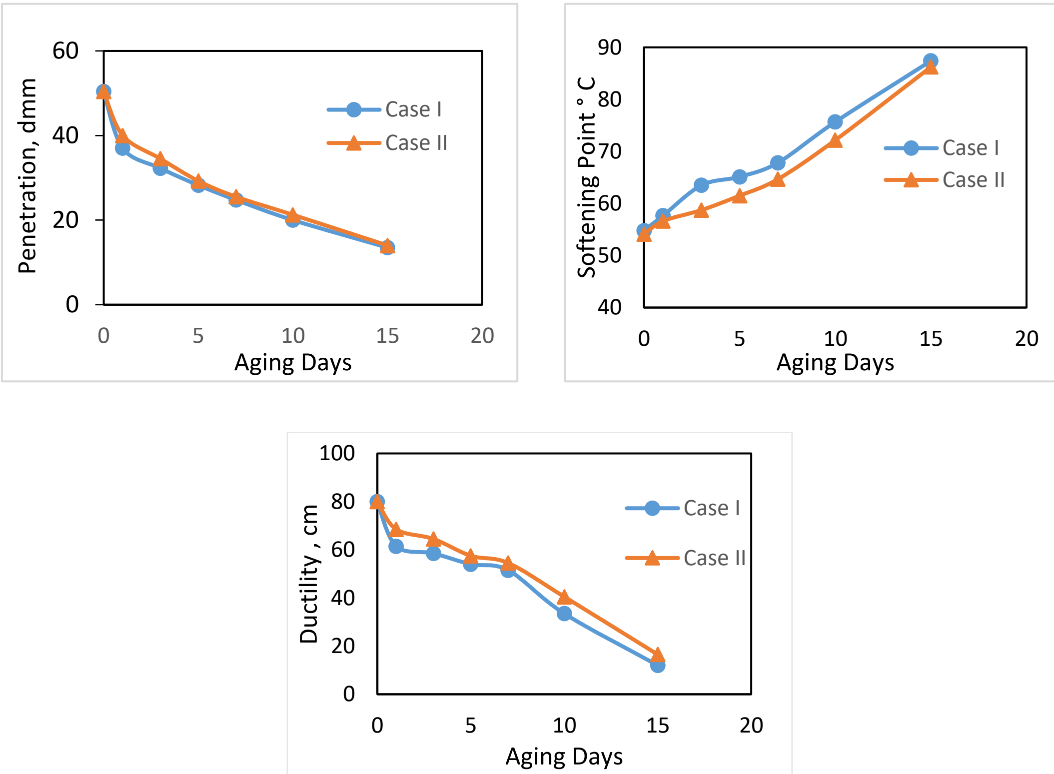


Figure 5.3 Variation in penetration, softening point and ductility values of Case I and Case II samples

It would be interesting to observe the diurnal variations in the changes of the properties and hence, the diurnal variations in all the above the properties were computed and presented in Table 5.6.

It can be seen clearly, from Table 5.6 that the changes in the properties were rapid during the first exposure (0-1-day exposure period) and then the properties kept changing more or less, at a gradual rate. The changes in properties are more with regard to Penetration values followed closely by the ductility values. However, the change in softening point was observed to be less rapid.

Table 5.6: Percentage Change per day in physical properties of binder

	Percentage Change (%) in the parameter (per day of oven exposure)					
Aging Period (days)	**In Penetration**		**In Softening**		**In Ductility**	
	Case I	**Case II**	**Case I**	**Case II**	**Case I**	**Case II**
0-1	-26.65	-20.70	5.23	4.68	-23.13	-14.38
1-3	-6.42	-6.88	5.06	1.85	-2.44	-2.92
3-5	-6.20	-7.61	1.29	2.36	-3.85	-5.43
5-7	-6.19	-6.41	2.07	2.57	-2.31	-2.61
7-10	-6.40	-5.56	3.85	3.87	-11.65	-8.56
10-15	-6.50	-6.82	3.10	3.90	-12.84	-11.85

The extracted binders from the field core samples were also subjected to the same set of physical investigations. The summary of the results obtained is presented in Table 5.7. From the Table 5.7, it is clear that the bitumen extracted from the top slices were aged more when compared with the bitumen extracted from the respective bottom slices. Also, the effects of aging in terms of reduced penetration, increased softening points and reduced ductility values were more prominent in the case of samples from NH 16, naturally aged for 7 years, when compared with the samples extracted from NH 65, naturally aged for 5 years. It is important to note that both these highways had more or less similar environmental variations and traffic exposure conditions. Also, a close observation of the values from bottom slices indicate that the aging effects are not completely absent, though lesser in comparison with the corresponding top slices.

Table 5.7 Physical properties of binder extracted from field cores

	Penetration (dmm)	Softening Point (° C)	Ductility (cm)
NH 65 Top	14.3	97.1	15.5
NH 65 Bottom	16.0	70.5	20.5
NH 16 Top	13.3	97.9	13.5
NH 16 Bottom	15.3	71.1	18.5

The ductility and penetration values of extracted binder from laboratory aged and field core samples has indicated good correlation (matching age is being mentioned in parenthesis) as shown in Table 5.8. Softening point results of the field extracted bitumen from top slices are high when compared with the bitumen extracted from laboratory aged samples, indicating that the artificial aging done, in the modified oven as per AASTHO R30, is not sufficient to fully simulate the field aging.

Table 5.8 Correlation of field aging with lab aging based on penetration and ductility

	Penetration (dmm)	**Softening Point (°C)**	**Ductility (cm)**
NH 65 Top	14.3 (14 days with Case I)	97.1 (oven aging is not enough)	15.5 (14 days with Case I)
NH 65 Bottom	16.0 (13 days for Case I)	70.5 (8 days for Case I)	20.5 (13 days for Case I)
NH 16 Top	13.3 (15 days)	97.9 (oven aging is not enough)	13.5 (15 days)
NH 16 Bottom	15.3 (14 days with Case I)	71.1 (8 days for Case I)	18.5 (14 days with Case I)

It can also be observed, from the values, presented in Table 5.8, that the top slice has aged more than the bottom slice by an equivalent of 1-day oven exposure in the laboratory. This reinforces the fact that the aging on the surface of the bituminous pavement will be more than the bottom layers. Changes in both ductility and penetration values also indicate the similar trend. However, the softening point showed a wide variation between the top and the bottom slices. This indicate that the laboratory aging with 5-day exposure as recommended by AASHTO R30 guidelines does not simulate the real field conditions fully.

5.3.2 Aging indices from fundamental properties of extracted binder

The perceivable changes in the basic properties of aged bitumen can provide critical clues regarding the extent of aging. Three different indices namely penetration retention percentage (PRP), penetration index (PI) and softening point increment (SPI) were calculated for all the samples as detailed below.

5.3.2.1 Penetration Retention Percentage (PRP)

Penetration Retention Percentage is defined as the ratio of penetration value of aged bitumen to that of penetration value of unaged bitumen. It can be expressed mathematically as equation 5.1:

$$PRP = \frac{\text{Penetration value of aged bitumen}}{\text{Penetration value of unaged bitumen}} \text{ X } 100 \quad \text{----------------- Eq. 5.1}$$

The PRP values of the bitumen extracted from Case I and Case II samples are presented in Table 5.9. For better understanding, the same PRP values are presented through Figure 5.4.

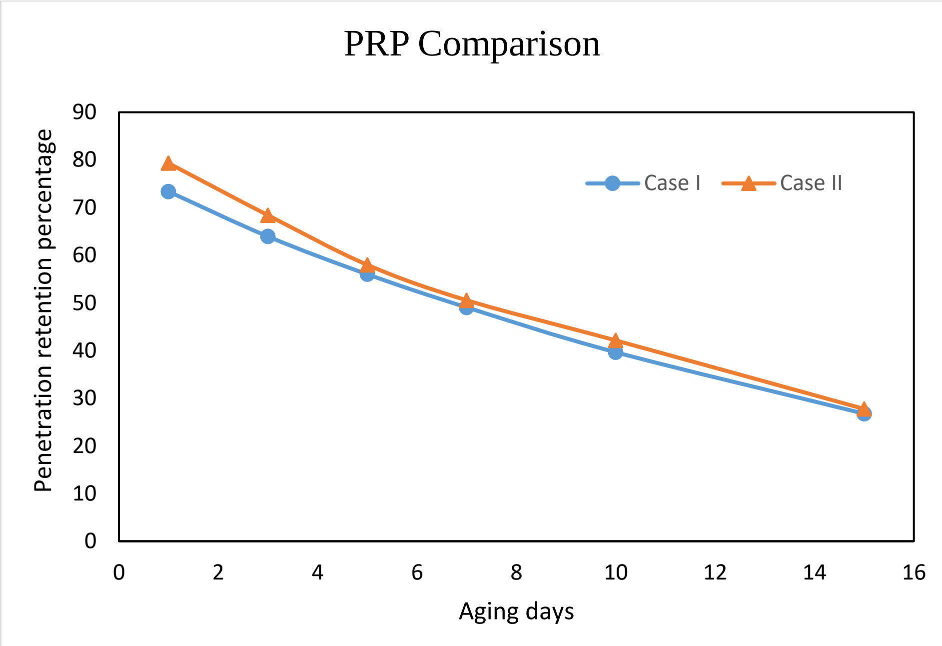


Figure 5.4. Comparison of PRP values for Case I and Case II samples

The PRP values, as observed in Figure 5.4 and Table 5.9, were gradually decreasing with incremental aging exposure. The PRP for the Case II binder was relatively higher at 79.30% after 1-day aging when compared with the PRP value observed for the unmodified Case I binder. As the aging progressed, the gap in PRP values of both modified and unmodified binders got considerably reduced. For instance, the PRP of Case I and Case II binders after 15 days of aging is 26.76% and 27.76% respectively with a very small difference between them.

The PRP values of bitumen extracted from the field cores were also calculated and summarized in Table 5.9. Comparative analysis between field and Case I laboratory based PRP values indicate the following.

- NH 65 top sample with PRP of 28.35% is equivalent with the PRP of 14.5-day aged Case I bitumen sample (From the correlation).
- NH 65 bottom sample with PRP of 31.72% is equivalent with the PRP of 13-day aged Case I bitumen sample (From the correlation).
- NH 16 top sample with PRP of 26.37% is equivalent with the PRP of 15-day aged Case I bitumen sample (From the correlation).

- NH 16 bottom sample with PRP of 30.33% is equivalent with the PRP of 13.5-day aged Case I bitumen sample (From the correlation).

The difference in aging of the bitumen extracted from the top and bottom slices in both the field cases was observed to be same as that of the 1.5-day laboratory exposure of the Case I laboratory samples. Also, it can be noted that the 7-year-old NH 16 cores (both top and bottom slices) have aged more than the corresponding samples of 5-year-old NH 65 cores by an equivalent half-a-day laboratory oven exposure of Case I samples.

5.3.2.2 Penetration Index (PI)

The penetration index values are calculated by following equation 5.2:

$$PI = \frac{1952 - 500 \log(\text{pen}) - 20 * SP}{50 \log(\text{pen}) - SP - 120} \quad \text{------------------- Eq. 5.2}$$

Where 'pen' is the penetration at 25 ° C and 'SP' is the softening point temperature.

As expected, the PI values, presented in Table 5.9 and Figure 5.5, were found to be increasing with aging exposure for both Case I and Case II samples. However, the Case II samples with additives have shown comparatively lesser PI vales when compared with the unmodified Case I samples, thus proving the efficacy of the additives being tried in the current study in abating the aging process.

However, the top slices of field samples have resulted in extremely high PI values when compared with laboratory aged samples. Thus, direct correlation could not be found between the laboratory aged samples with those, collected from the field cores. The PI of the bitumen extracted from the bottom slice is very low when compared with those values obtained for bitumen from corresponding top slices.

Table 5.9 Penetration test results and analysis of extracted binder

Aging (Exposure in Modified Oven)	Penetration (dmm)		Penetration retention percentage (PRP)		Penetration Index (PI)	
Days	**Case I**	**Case II**	**Case I**	**Case II**	**Case I**	**Case II**
0	50.44	50.44	NA	NA	NA	NA
1	37.00	40.00	73.35	79.30	-0.15	-0.19
3	32.25	34.50	63.94	68.40	0.67	-0.08
5	28.25	29.25	56.01	57.99	0.68	0.10
7	24.75	25.50	49.07	50.56	0.86	0.39
10	20.00	21.25	39.65	42.13	1.62	1.22
15	13.50	14.00	26.76	27.76	2.39	2.31
	Penetration (dmm)		**PRP With reference to penetration of 50.44 dmm**		**Penetration Index (PI)**	
NH 65 Top	14.30		28.35		3.52	
NH 65 Bottom	16.00		31.72		0.48	
NH 16 Top	13.30		26.37		3.56	
NH 16 Bottom	15.30		30.33		0.46	

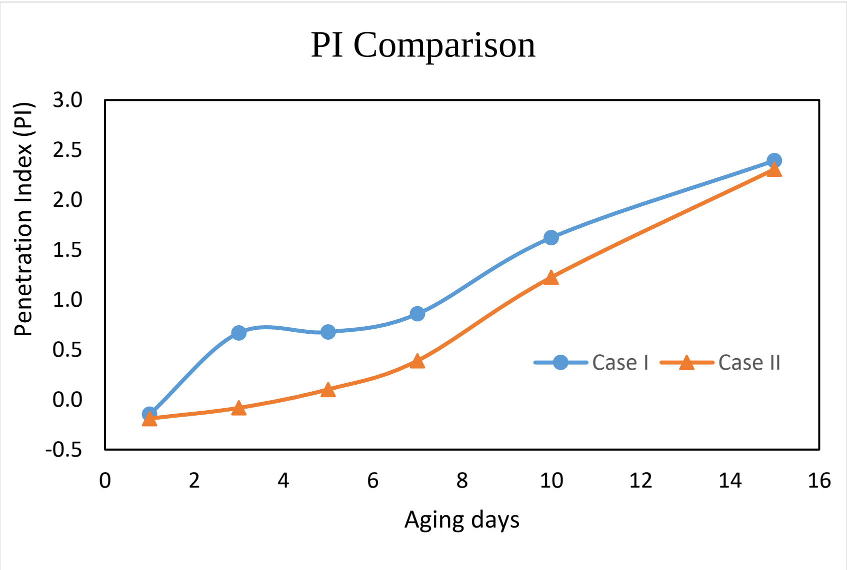


Figure 5.5 Comparison of PI values for Case I and Case II samples

5.3.2.3 Softening Point Increment (SPI)

The Softening Point Increment (SPI) is another possible indicator for estimating the degree of aging that occurs in the bitumen binders. It is obtained by the following formula

SPI = softening point temperature of the aged binder - softening point temperature of unaged binder

The SPI values for bitumen extracted from Case I and Case II samples is presented through Table 5.10 and Figure 5.6.

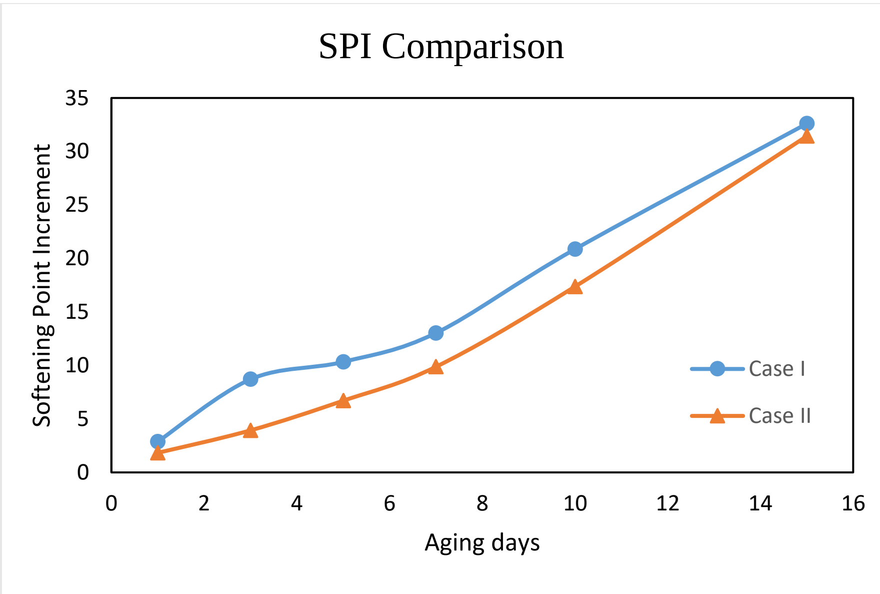


Figure 5.6 Comparison of SPI values for Case I and Case II samples

The SPI values for both Case I and Case II samples have shown growing trend with progressive aging in general. However, for the Case II modified sample, the trend is smoother than for the sample without any modifications.

The SPI values of the top slices for both NH 65 and NH 16 were observed to be 42.3 and 43.1 respectively. These values were found to be much higher than that obtained from the laboratory 15-day-aged Case I sample (32.6), indicating that the 15-day exposure may not be enough to simulate the field aging (top slice) as per this index. However, the SPI values of the bottom slices of both National Highways closely match with that of 9-day aged Case I sample.

Table 5.10 Softening point test results and analysis of extracted binder

Aging (Exposure in Modified Oven)	**Softening Point (° C)**		**Softening Point Increment (SPI)**	
Days	**Case I**	**Case II**	**Case I**	**Case II**
0	54.80	54.80	NA	NA
1	57.67	56.63	2.87	1.83
3	63.50	58.73	8.70	3.93
5	65.13	61.50	10.33	6.70
7	67.83	64.67	13.03	9.87
10	75.67	72.17	20.87	17.3
15	87.40	86.23	32.60	31.43
	Softening Point (° C)		**Softening Point Increment (SPI)**	
NH 65 Top	97.10		42.3	
NH 65 Bottom	70.50		15.7	
NH 16 Top	97.90		43.1	
NH 16 Bottom	71.10		16.3	

5.3.3 Rheological studies on extracted bitumen from laboratory and field aged specimens

5.3.3.1 Viscosity by Brookfield's Rotational Viscometer

The changes in kinematic viscosity of the aged bitumen, when compared with the virgin bitumen, are expected to provide insight into the physical changes happening with aging. It is with this premise that the kinematic viscosities were found for field aged bitumen samples and the laboratory aged samples. Brookfield's rotational viscometer was employed for this test in accordance with ASTM D4402/ D4402M-15.

The bitumen samples, recovered from laboratory aged and field samples, were subjected to kinematic viscosity tests in the temperature range of 120 ° C to 190 ° C. The results of Case I and Case II samples were presented in Figures 5.7 and 5.8. The viscosities of field extracted samples were presented along with the Case I and Case II samples with 0 and 15-day exposures for comparison, through Figure 5.9.

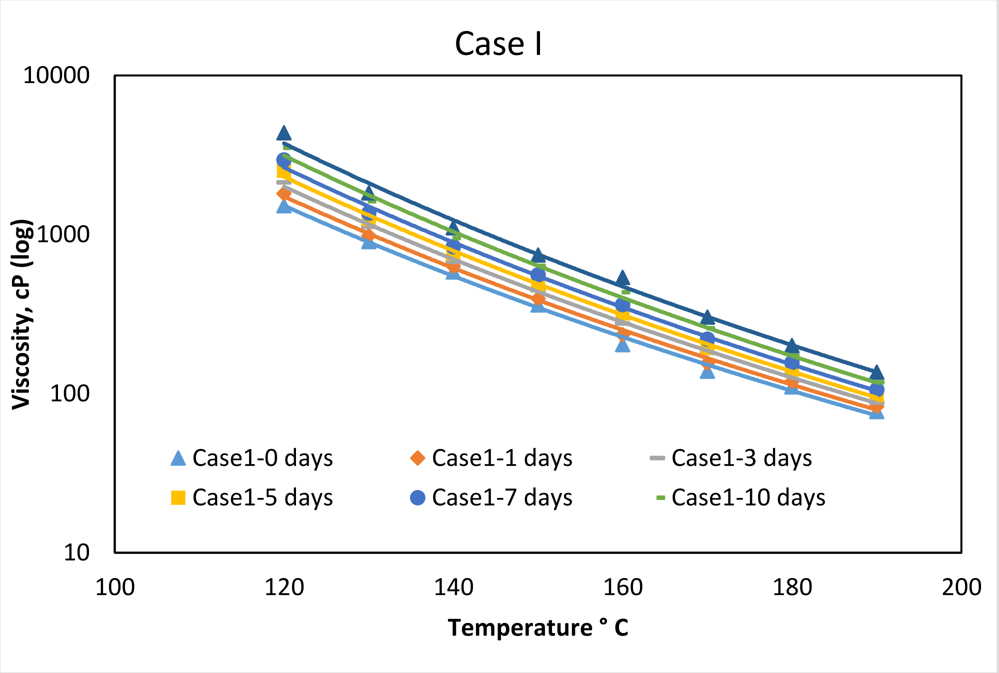


Figure 5.7 Viscosity – Temperature variations for Case I bitumen binders

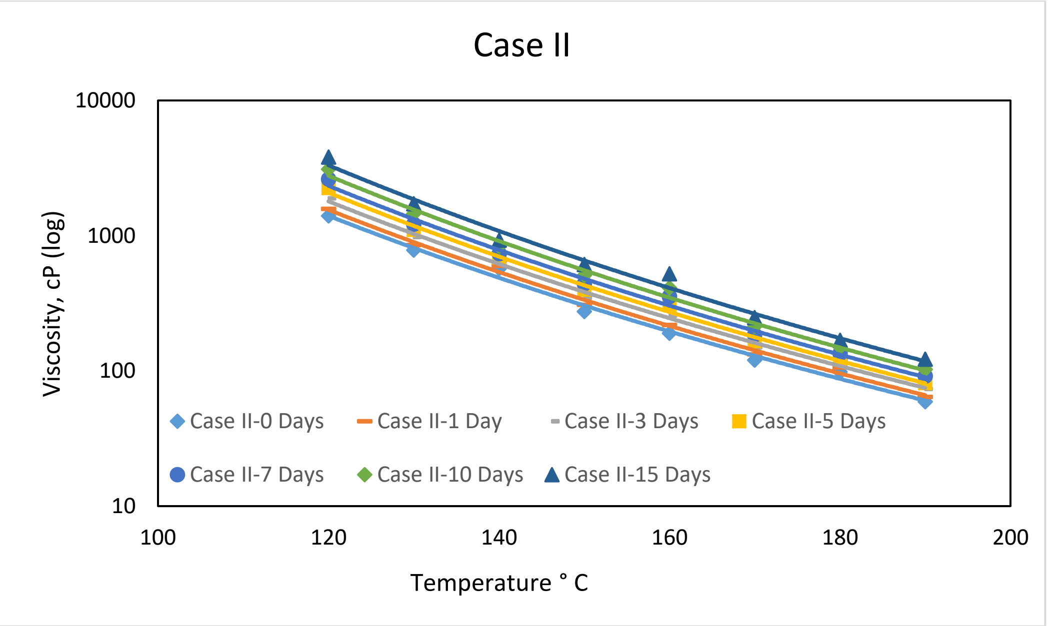


Figure 5.8 Viscosity – Temperature variations for Case II bitumen binders

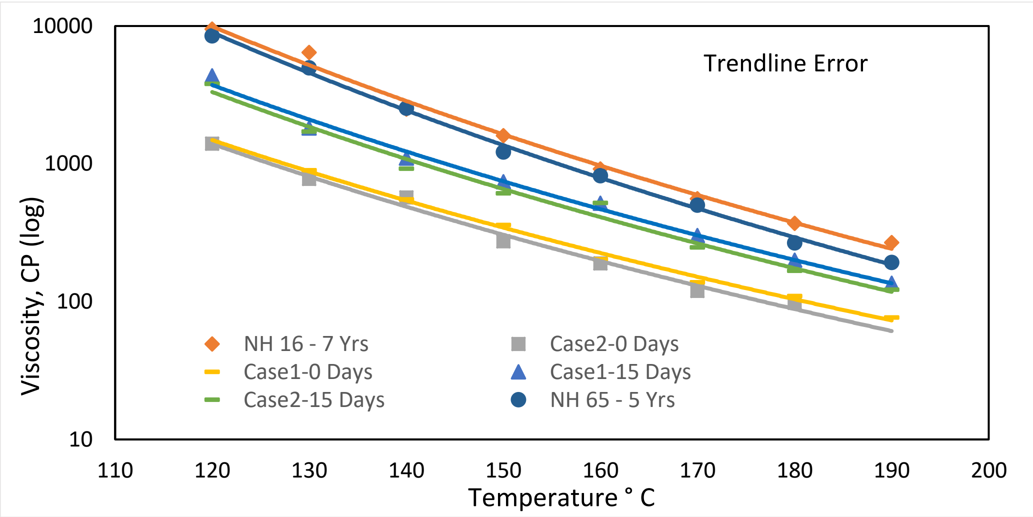


Figure 5.9 Viscosity – Temperature relationship for field and lab aged bitumen binders

From the graphs, it can be clearly seen that

- The temperature viscosity relation shown that the bitumen extracted from Case II specimens exhibited the Newtonian behavior in the temperature range of 120 ° C to 190 ° C and it remained non-Newtonian below 120 ° C. This could be because of the Sasobit used SX105 had a melting point of 105 ° C.
- From the study it is clear that the Sasobit will help as an additive in improving the workability of the mixture in the temperature range of 130 ° C to 140 ° C and only when extracted binder was tested the affect of Sasobit was found to be marginal with respect to the reduction in viscosity. However, the hydrated lime was added as an antioxidant filler and hence its affect was not seen on the extracted binder.
- The expected trend of enhancement in the viscosities with aging is clearly seen during the current experimentation. The extracted binder, with and with out Sasobit showed the same trend.

5.3.3.2 Dynamic Shear Rheometer (DSR) results and aging indices of extracted bitumen from laboratory and field aged specimens

During the current research activity, comprehensive rheological investigations were carried out using Anton Paar's Dynamic Shear Rheometer (DSR). The linear amplitude sweep tests were performed under controlled strain mode and the properties namely complex modulus (G*) and phase angle (δ) were found for both the laboratory and field aged bitumen samples. The aging indices based on these results are evaluated and presented in the following sections.

The complex shear modulus values of binder extracted from Case I and Case II samples aged for different periods of time are presented in Figure 5.10 and Figure 5.11 respectively. From these Figures, it can be seen that the complex shear modulus values of Case I samples are more than those of Case II samples for respective aging periods.

It can be observed that the rate of aging that occurred in the Case II samples is uniform (as the curves of different aging periods are almost parallel and equidistant as seen in Figure 5.11) when compared with Case I samples. Also, it can be seen that the rate of aging between 10 and 15 days is more for Case I samples when compared with the Case II samples. These observations show that the addition of hydrated lime and Sasobit to the bituminous mixtures has considerably improved the aging resistance.

The complex shear modulus values of bitumen extracted from top and bottom slices of NH 65 and NH 16 are presented in Figure 5.12 along with virgin binder. From the Figures 5.10, 5.11 and 5.12, it can be noted that the complex shear modulus values of bitumen extracted from NH 65 bottom slice are almost equal to the values of 10 days aged Case I and Case II samples. The complex shear modulus values of NH 65 top slice are almost equal to 15 days aged Case I samples and more than 15 days aged Case II samples.

The complex shear modulus values of NH 16 bottom slice are in between 10 and 15 days aged Case I and Case II samples. The values of NH 16 top slice are almost equal to the complex shear modulus values of corresponding to 15 days aged Case I and Case II samples.

This clearly shows that the level of aging that occurred in NH 65 (five years in-service) is almost equal to the 15-day aging simulated in laboratory but for NH 16 (Seven year in service) the level of aging that occurred in actual field conditions is more, and thus requires more rigorous artificial aging.

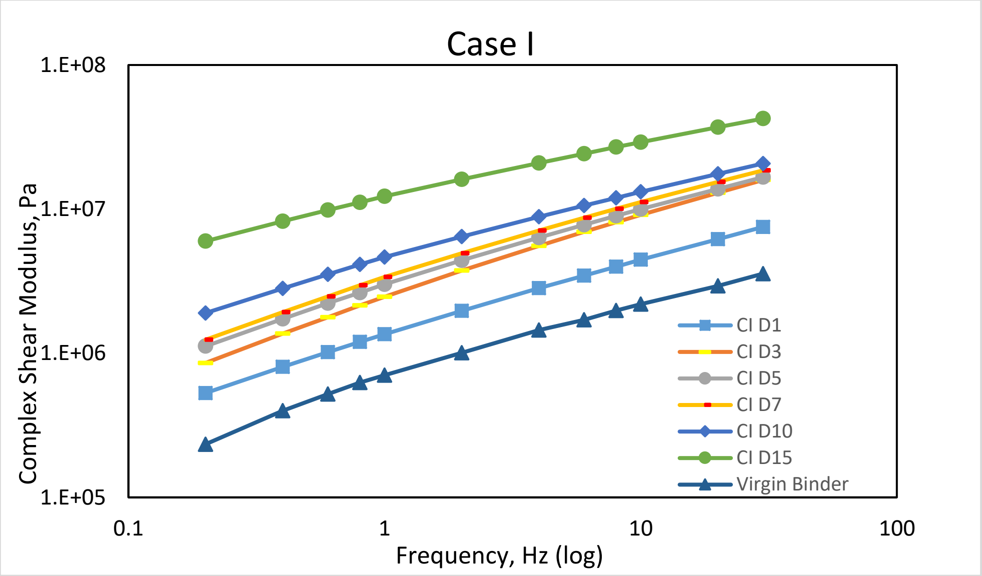


Figure 5.10 Complex shear modulus values after aging for Case I samples

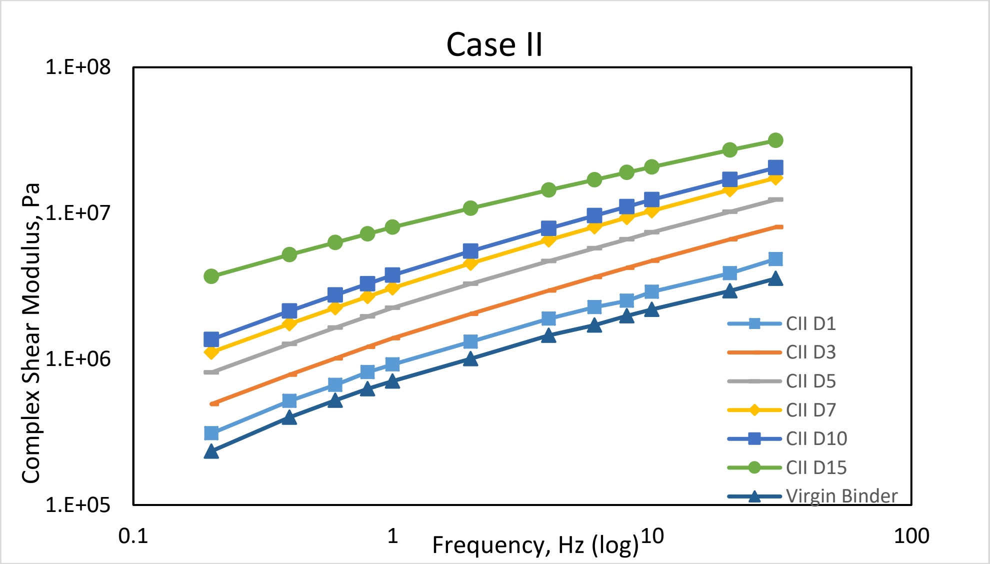


Figure 5.11 Complex shear modulus values after aging for Case II samples

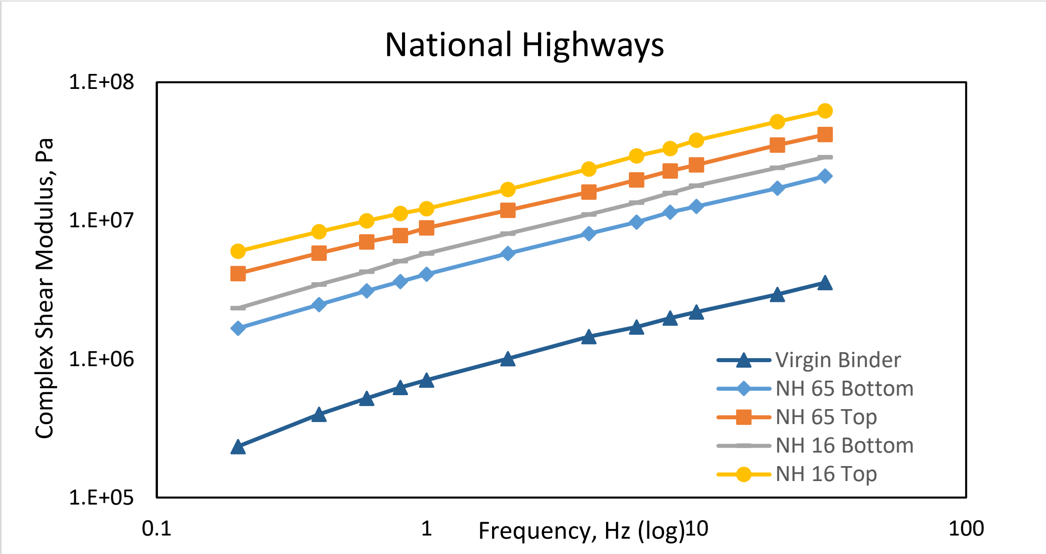


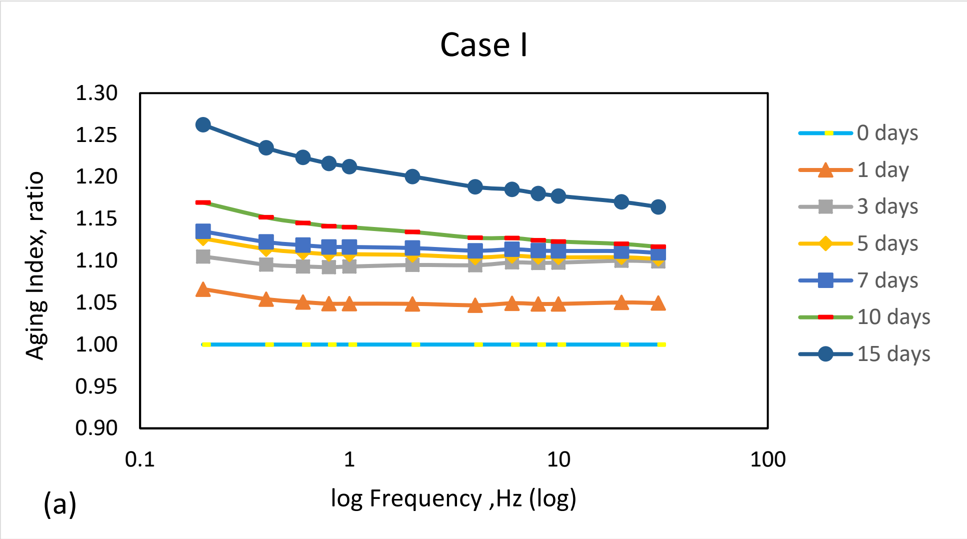
Figure 5.12 Complex shear modulus values after aging for National highways

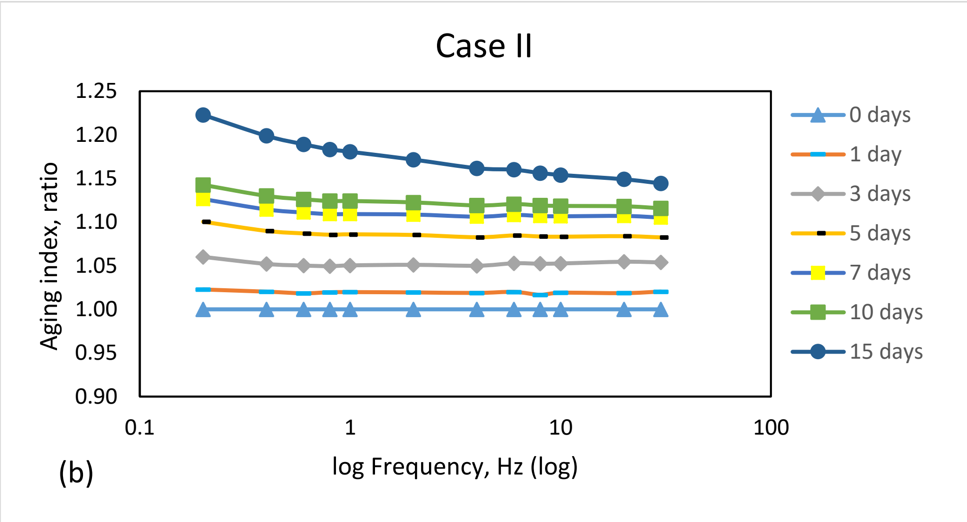
5.3.3.2.1 Aging index using complex shear modulus (G*)

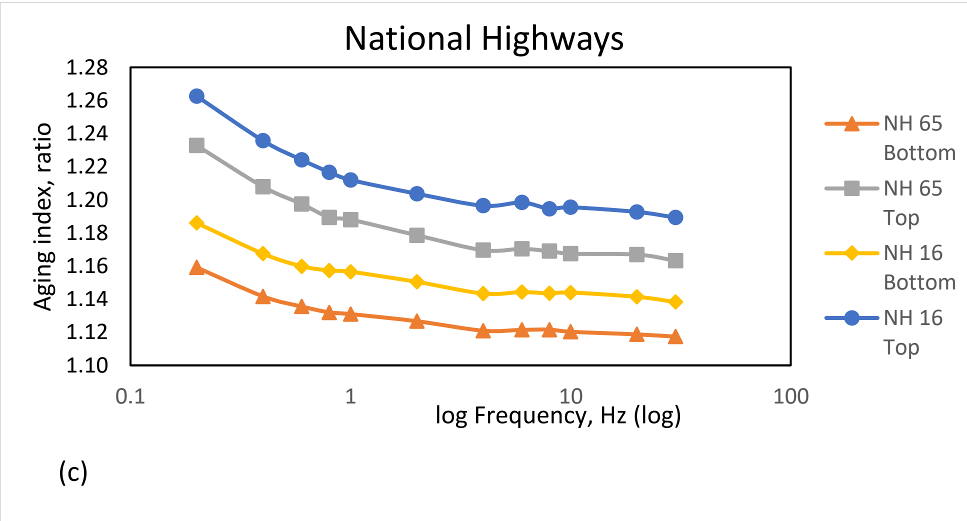
Aging index based on complex shear modulus is computed for all the aged samples with the following equation 5.3, suggested by Ongel et. al. (2014)

$$\frac{\log G* \text{ (aged)}}{\log G* \text{ (unaged)}} \text{ ----------------- Eq.5.3}$$

Aging indices are computed for all the aging regimes and presented through Figures 5.13 (a), 5.13 (b) and 5.13 (c) for Case I, Case II and field samples respectively. Frequencies from 0.2 Hz to 30 Hz were considered during the experimentation and the same has been presented. Representative aging index is worked out by averaging the indices for each of the aging regimes and the results are shown in Figure 5.13 (d) for further use.


Case I
Aging Index, ratio
1.30
1.25
1.20
1.15
1.10
1.05
1.00
0.95
0.90
0.1
1
10
100
log Frequency ,Hz (log)
0 days
1 day
3 days
5 days
7 days
10 days
15 days
(a)


Case II
Aging index, ratio
1.25
1.20
1.15
1.10
1.05
1.00
0.95
0.90
0.1
1
10
100
log Frequency, Hz (log)
0 days
1 day
3 days
5 days
7 days
10 days
15 days
(b)


National Highways
Aging index, ratio
1.28
1.26
1.24
1.22
1.20
1.18
1.16
1.14
1.12
1.10
0.1
1
10
100
log Frequency, Hz (log)
NH 65 Bottom
NH 65 Top
NH 16 Bottom
NH 16 Top
(c)

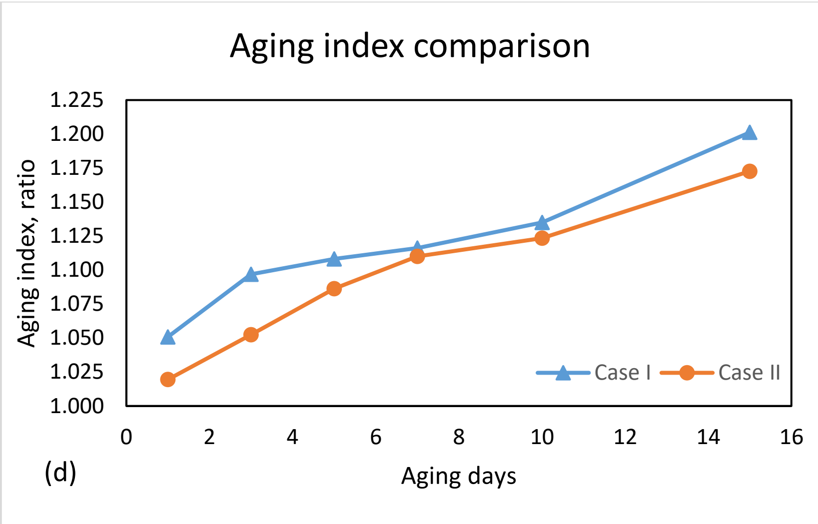


Figure 5.13. G* based aging index ratio for (a) Case I (b) Case II (c) National Highways (d) representative indices for different stages of aging

From Figures 5.13 (a) to 5.13 (c), it can be observed that the Case I samples have shown more aging indices when compared with corresponding aging indices observed for Case II samples, thus showing the clear influence of additives in abating the aging phenomenon. This observation is true for all the aging regimes (1 day to 15 days), as can be seen from the Figure 5.13. Representative aging indices presented in Figure 5.13 (d) also strengthens the viewpoint that the Case I samples, without additives, have shown more aggressive aging when compared with Case II samples having the Sasobit and hydrated lime as additives.

With a view to make a comparison between field aging and laboratory aging processes, the average aging indices, calculated for the field samples, were matched with the laboratory aging indices available for Case I samples from Figure 5.13 (d). The results of this analysis are presented through Table 5.11 for ready reference.

Table 5.11 Correlation between field and laboratory aged bitumen samples using complex shear modulus based aging index

	Avg. Aging Index based on complex shear modulus	Matching age in days of oven exposure of Case I samples	Matching age in days of oven exposure of Case I samples
NH 65 Top	1.183	14	Aging not enough
NH 16 Top	1.210	Aging not enough	Aging not enough
NH 65 Bottom	1.129	10	11
NH 16 Bottom	1.153	11	13

From the Table 5.11, where complex shear modulus based aging indices are presented, it can be observed that 14 days of oven aging was needed to simulate the field aging occurred in the top slice of NH 65, which was field aged for 5 years. The corresponding bottom slices of the samples collected from the same site needed 10 days of oven exposure.

The top slice of 7-year aged NH 16 samples could not be simulated fully even with the 15 days of extended oven exposure while the bottom slices of the corresponding cores needed 11 days of modified oven exposure for simulation.

5.3.3.2.2 Aging index using fatigue resistance parameter - G* sin (δ)

It is a well-known fact that the fatigue cracking accelerates with advanced aging and is usually governed by G* sin (δ). Hence, it was felt that an aging index based on G* sin (δ) could explain the aging process more accurately. It was with this premise that the G* sin (δ) based aging index was used to understand the level of aging happening in the laboratory and compare the same with the naturally aged field samples.

G* sin (δ) based aging index can be calculated as equation 5.4

$$\text{G* sin (δ) based aging index} = \frac{(G* \sin(\delta))aged}{(G* \sin(\delta))unaged} \quad \text{----------------- Eq. 5.4}$$

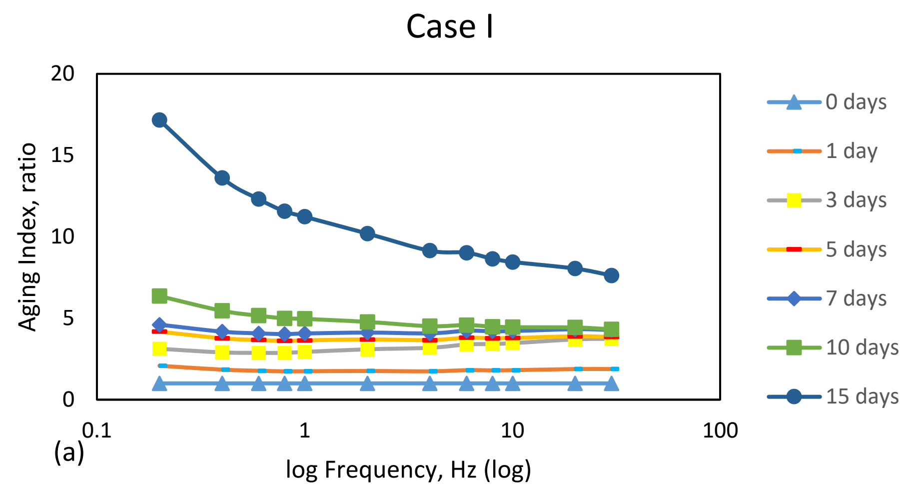
The aging index values using fatigue resistance parameter with respect to frequency for Case I, Case II and field extracted bitumen samples are presented in Figure 5.14 (a), 5.14 (b) and 5.14 (c) respectively. From these Figures, it can be concluded that the fatigue resistance keeps on decreasing with the progression of aging as the aging index values are high for 15-day aged samples when compared with the lesser aged samples for both the cases.

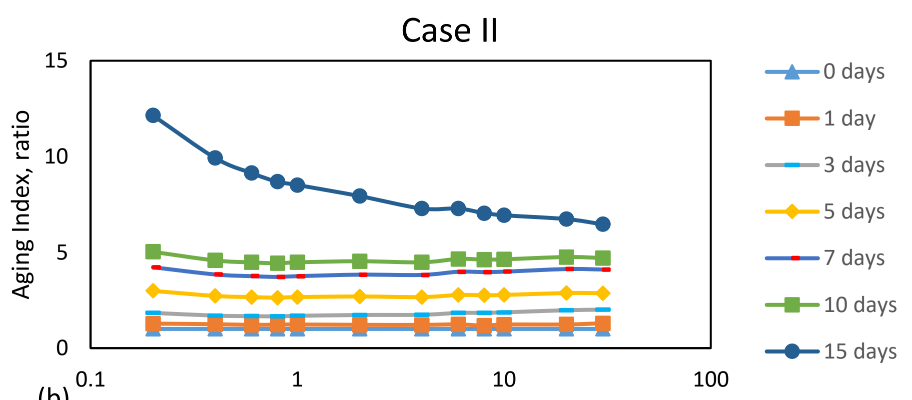
The Figure 5.14 (d) shows the comparison of Case I and Case II average aging indices developed using fatigue resistance parameter, G* sin (δ), with respect to aging periods. One important observation, deduced from the Figure, is that the aging index values are almost same for both cases from 7 to 10 days of aging and from the Figure 5.14 (d), it can be seen that the fatigue resistance is less for Case I samples that are prepared without any additives, when compared with Case II samples.

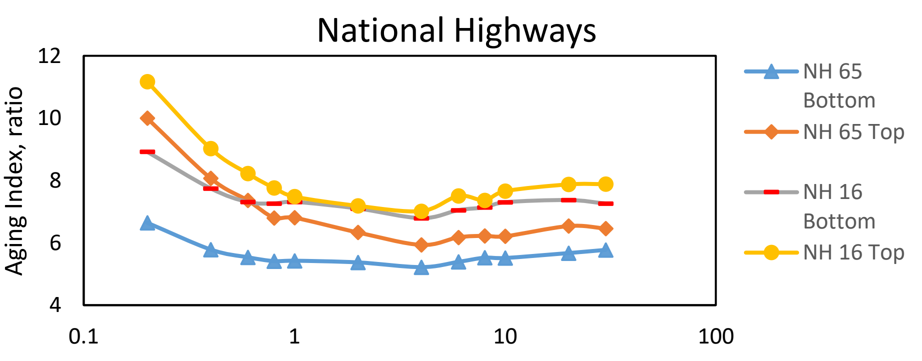
Table 5.12 and Figure 5.14 (d) shows the correlation between the field aging and laboratory aging by using aging index developed from fatigue resistance parameter. From the Table 5.13, it can be deduced that the amount of aging that occurred in bitumen extracted from NH 65 bottom slice is equal to 11 days aging of Case I samples and 12 days aging of Case II samples. For the top slice of the core extracted from NH 65, aging occurred is equivalent to the 12-day aged sample of Case I and 14-day aged sample of Case II. For the bitumen extracted from NH 16 bottom slice, the level of aging is matched with the equivalent 12- day laboratory aging for Case I samples and 14-day laboratory aging for Case II samples. The degree of aging in NH 16 top slice is observed to be equivalent to that of 13-day aged sample of Case I and 15-day aged sample for Case II samples.

Table 5.12 Correlation between field and laboratory aged samples using fatigue resistance parameter

	Avg. Aging Index based on G* sin (δ)	**Case I (Days)**	**Case II (Days)**
NH 65 Bottom	5.61	11	12
NH 65 Top	6.91	12	14
NH 16 Bottom	7.38	12	14
NH 16 Top	8.01	13	15


Case I
Aging Index, ratio
20
15
10
5
0
0.1
1
10
100
log Frequency, Hz (log)
0 days
1 day
3 days
5 days
7 days
10 days
15 days
(a)


Case II
Aging Index, ratio
15
10
5
0
0.1
1
10
100
log Frequency, Hz (log)
0 days
1 day
3 days
5 days
7 days
10 days
15 days
(b)


National Highways
Aging Index, ratio
12
10
8
6
4
0.1
1
10
100
log Frequency, Hz (log)
NH 65 Bottom
NH 65 Top
NH 16 Bottom
NH 16 Top
(c)

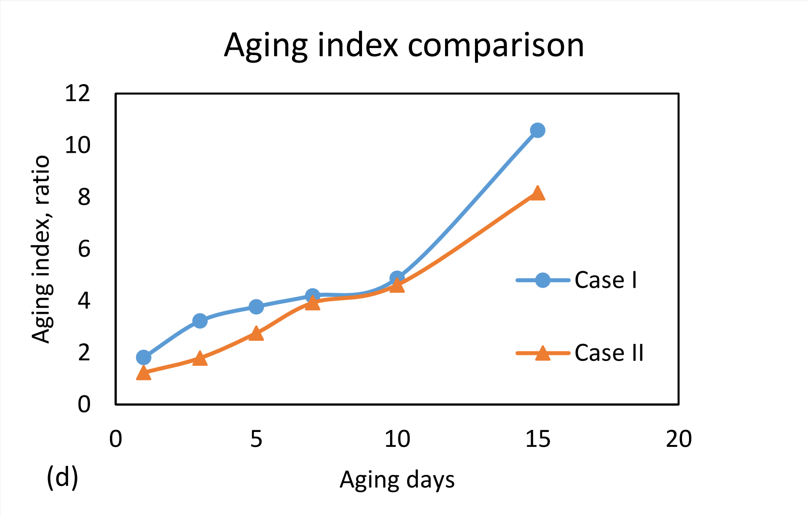


Figure 5.14. Aging index ratio based on G*sin (δ) for (a) Case I (b) Case II (c) National Highways (d) aging index comparison of G*sin (δ) for Case I and Case II

5.3.3.2.3 Aging index using loss tangent (tan (δ))

The aging index ratio of loss tangent is defined as the ratio of aged tan (δ) to unaged tan (δ) of extracted bitumen binder, which can be expressed mathematically as equation 5.5:

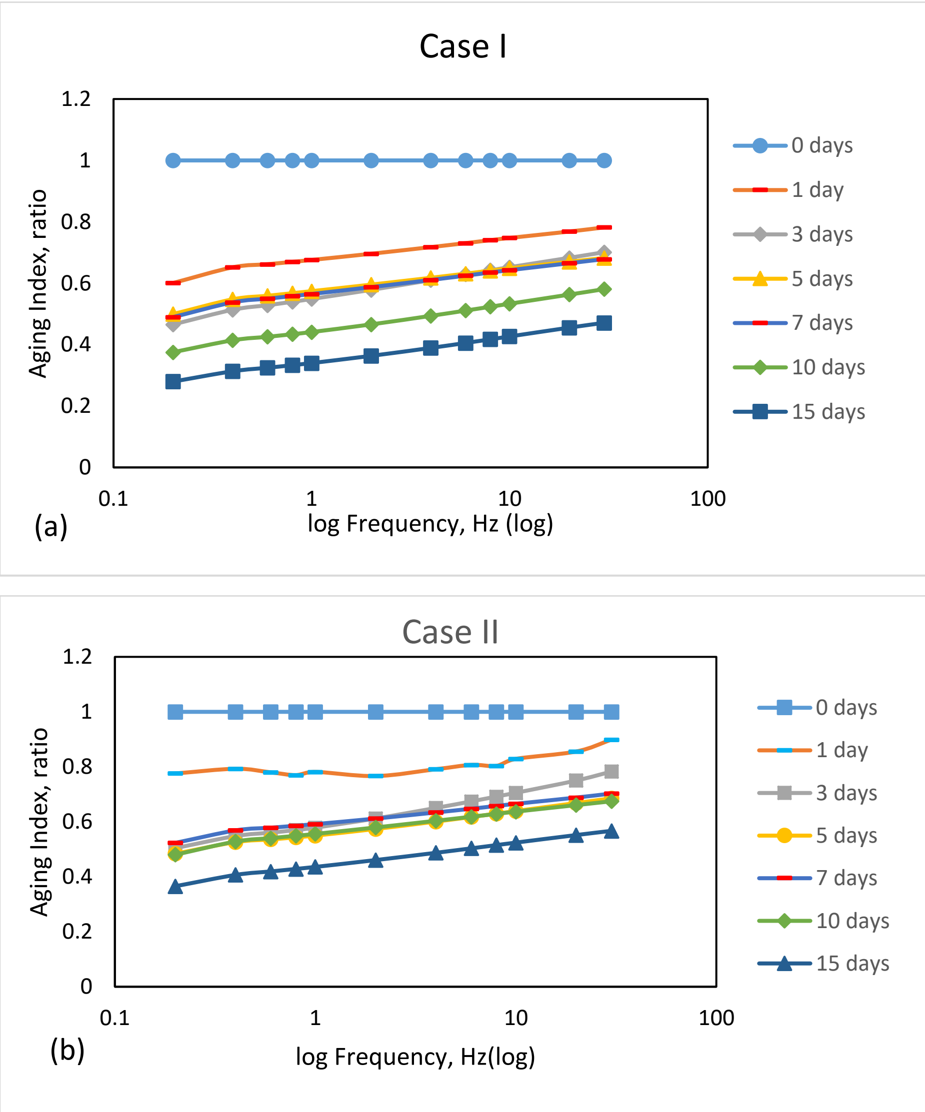
$$\text{Aging index using loss tangent} = \frac{\tan(\delta)\text{aged}}{\tan(\delta)\text{unaged}} \quad \text{------------------ Eq. 5.5}$$

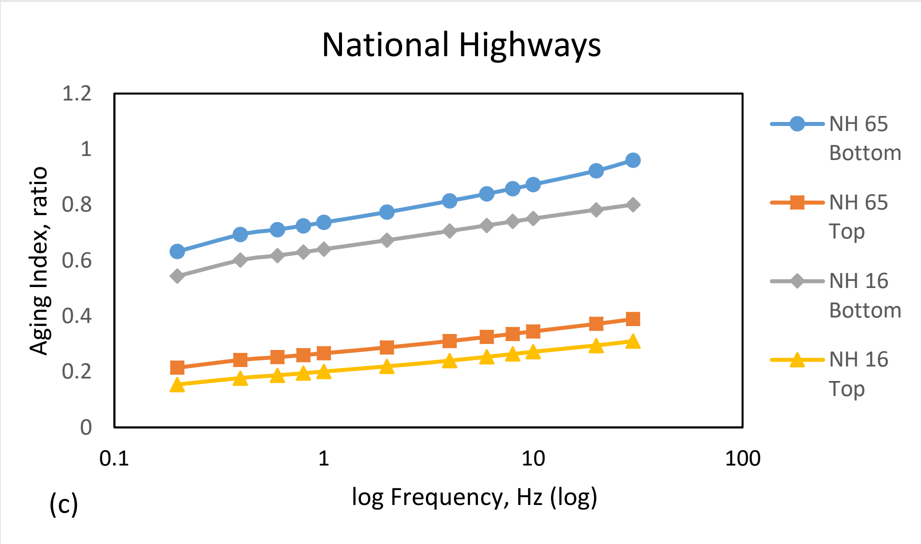
The plots of aging index using loss tangent against frequency for Case I, Case II and field bitumen samples are presented in Figure 5.15 (a), 5.15 (b) and 5.15 (c) respectively. It can be noted that the aging index values keep reducing with progressive aging for the corresponding frequencies, for both Case I and Case II samples and also for top to bottom slices of field samples. From the Figure 5.15, it can be observed that the rate of aging is more pronounced within the first day of aging for Case I samples when compared with the Case II samples with additives.

Form Figure 5.15 (c), it can be noted that, the bottom slices of the both national highways retained more elasticity when compared with the corresponding top slices.

This again reinforces the notion that the top layers will age more aggressively when compared with the corresponding bottom layers of the bituminous concrete pavements.

The Figure 5.15 (d) shows the comparison between the aging indices of Case I and Case II bitumen samples with the duration of aging. From the Figure, it can be observed that the aging indices of Case II bitumen samples with additives are more when compared with the Case I bitumen samples without additives. The higher value of tan (δ) aging index is attributed to higher elastic behavior indicating the increased retention of elastic behavior in Case II samples when compared with Case I bitumen samples. This observation again proves the efficacy of Sasobit and hydrated lime in reducing the adverse effects of aging in bituminous concrete mixtures.





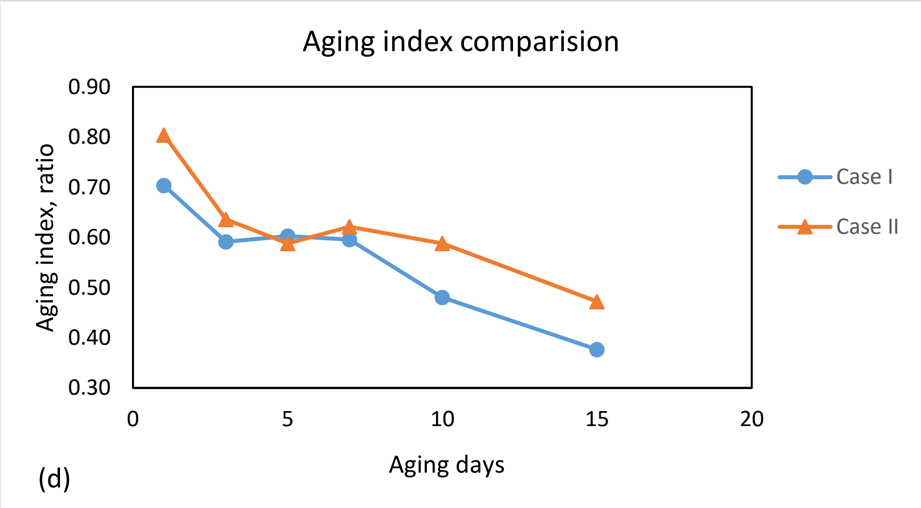


Figure 5.15. **Aging index ratio of tan (δ) for (a) Case I (b) Case II (c) National Highways (d) aging index comparison of tan(δ) for Case I and Case II**

5.3.3.3 Multiple Stress Creep Recovery (MSCR) Test on extracted bitumen from laboratory and field aged specimens

Multiple Stress Creep Recovery (MSCR) test is an improvement over the high-temperature Super Pave binder grading specifications, and it is performed in accordance with AASHTO TP70 (Soenen et al. 2013). The test is used to evaluate the rutting parameter of the binder by recording the per cent recovery (R %) and non-recoverable creep compliance per unit shear stress (J_{nr}) at two stress levels of 0.1 kPa and 3.2 kPa. The J_{nr} is determined by recording the non-recoverable strain and dividing it by the stress applied (0.1 kPa or 3.2 kPa). These two parameters, namely R% and J_{nr}, serve as an indicator of the mixture's ability to be resilient under heavy loading which leads to permanent deformation. Based on the J_{nr} and $J_{nr-diff}$. values, the grading of the binder is assigned.

The extracted bitumen samples were subjected to Multiple Stress Creep Recovery (MSCR) testing as per AASHTO TP70 (2013). The non-recoverable creep compliance (J_{nr}) values were measured at the standard temperature of 64 ^{0} C. Samples having dimensions of 1 mm thickness and 25 mm diameter, were subjected to a creep load for a duration of 1 s followed by a recovery duration of 9 s under standard stress levels of 0.1 kPa and 3.2 kPa. The samples were subjected to 10 cycles each of loading and unloading at both the stress levels. To evaluate the stress sensitivity towards varied loading conditions, $J_{nr-diff}$ was also computed. A summary of J_{nr} at both the stress levels along with $J_{nr-diff}$ for all the laboratory (both Case I and Case II) and field aged samples is summarized and presented in Table 5.13. J_{nr} and $J_{nr-diff}$ are calculated using the following equations 5.6 and 5.7:

$$J_{nr}(\tau) = \frac{\sum_{i=1}^{10}\left(\frac{e_{10}}{\tau}\right)_i}{10} \quad \text{------------------------- Eq. 5.6}$$

$$J_{nr\_diff}(\%) = \frac{(Jnr @ 3.2) - (Jnr @ 0.1)}{(Jnr @ 0.1)} \times 100 \quad \text{----------------- Eq. 5.7}$$

Where,

J_{nr} (τ) = percentage of avg. non recoverable creep compliance at stress level (τ)

τ = standard stress level of 0.1 and 3.2 kPa.

Table 5.13 Comparison of J_{nr} values of aged Case I and Case II and field samples

	Aging, Days	J_{nr} @ 0.1 kPa	J_{nr} @ 3.2 kPa	J_{nr_diff} (%)	Stress Sensitivity Criteria Satisfied (Yes or No)
Case I	0	2.616	3.187	21.82	Yes
	1	2.117	2.514	18.74	Yes
	3	1.519	1.802	18.62	Yes
	5	1.421	1.670	17.51	Yes
	7	0.772	0.89	16.27	Yes
	10	0.374	0.425	13.61	Yes
	15	0.171	0.193	13.00	Yes
Case II	0	2.616	3.187	21.82	Yes
	1	2.238	2.664	19.03	Yes
	3	1.631	1.941	18.99	Yes
	5	1.531	1.806	17.92	Yes
	7	0.885	1.036	17.02	Yes
	10	0.484	0.556	14.85	Yes
	15	0.182	0.208	13.96	Yes
NH 65 Top	5 years	0.191	0.211	10.67	Yes
NH 65 Bottom	5 years	1.394	1.599	14.66	Yes
NH 16 Top	7 years	0.099	0.107	7.31	Yes
NH 16 Bottom	7 years	1.243	1.417	14.02	Yes

From the Table 5.13 it can be observed that the J_{nr} and $J_{nr\text{-}diff}$ values, at both 0.1 kPa and 3.2 kPa loading, are decreasing with respect to aging for both Case I and Case II samples. The values of J_{nr} are higher for Case II samples at both loadings when compared with the Case I samples. The lower J_{nr} values of Case I samples indicate that the bitumen extracted from these samples is stiffer and has undergone more aging when compared with the Case II samples (Das, P.K.et al. 2017, Singh et al. 2019). The higher J_{nr} values of Case II samples clearly shows the anti-aging effect of Sasobit and hydrated lime on the bitumen at both high and low stress levels.

The J_{nr} values of 7-year-old NH 16 are less than those of 5-year-old NH 65 indicating the excessive aging occurred in NH16. It can be observed that the J_{nr} values of the bitumen extracted from top portion of the pavements are less than those of the bitumen extracted from bottom portion, once again confirming that the top portion undergoes more aging when compared with the bottom portion of the pavement.

When the J_{nr} values are compared for the NH 65, NH 16, Case I and Case II samples, it can be noted that around 14 days of laboratory oven aging is necessary to simulate the aging occurred in NH 65 top portion. It can also be noted that the artificial laboratory oven aging is not enough to simulate the actual aging that occurred in the NH 16 specimens.

5.3.4 Fourier Transform Infrared (FT-IR) Spectroscopy results of extracted bitumen from laboratory and field aged specimens

It is a well-established fact that the oxidation is main cause of aging mechanism. Oxidation can be measured quantitatively by functional group analysis by employing FT-IR. The absorbance bands at 1030 cm $^{-1}$ are formed because of S=O stretch in sulfoxides and the absorbance bands at 1700 cm $^{-1}$ are formed because of C=O stretch in carbonyl compounds. The peaks associated with these two wave numbers can be attributed to the presence of sulfoxides and carbonyl compounds.

The FT-IR spectrums for Case I, Case II and field samples are presented through Figures 5.16, 5.17 and 5.18. The 15-day laboratory modified oven aged sample has shown highest peak at the absorbance bands 1030 cm $^{-1}$ and 1700 cm $^{-1}$, when compared with other peaks with lesser aging exposure, thus proving the analogy that the peaks go up with aging. It can also be seen that the absorbance values for Case II (with hydrated lime and Sasobit addition) sample, having the similar aging exposure of 15 days, has shown lesser values when compared with the corresponding sample belonging to Case I. This clearly demonstrated the efficacy of the addition of hydrated lime and warm mix additive, Sasobit, in bitumen. It can be concluded that the hydrated lime with its anti-aging and anti-stripping properties, has prolonged the life of bitumen. Also, the warm mix additive, Sasobit had reduced the initial heating requirement while preparing the mixture, there by contributing towards reduced short term aging.

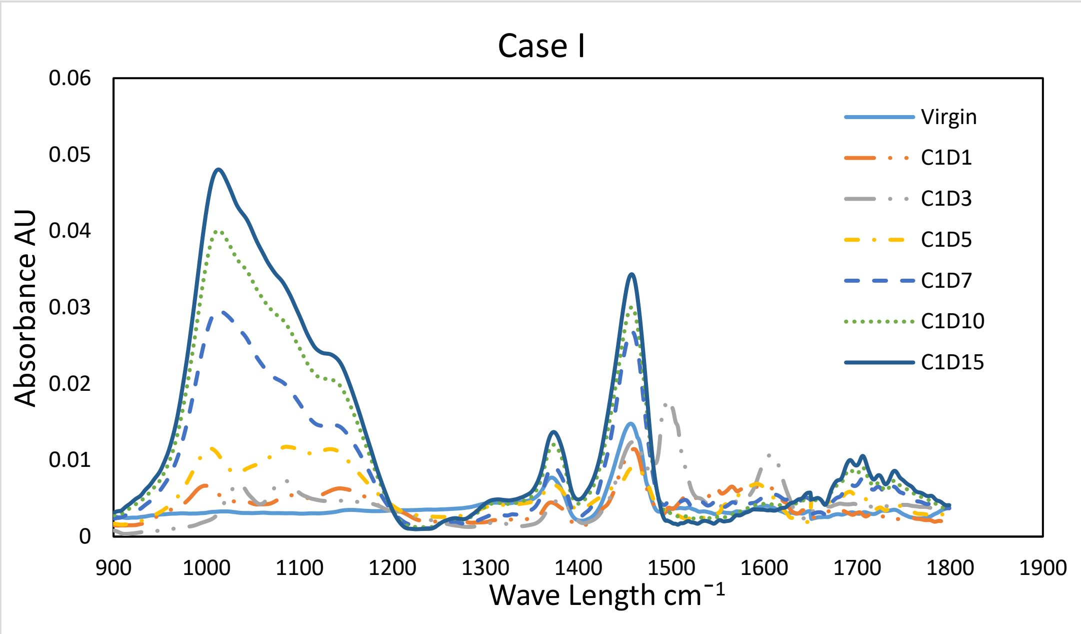


Figure 5.16 FT-IR spectrum of bitumen extracted from Case I samples

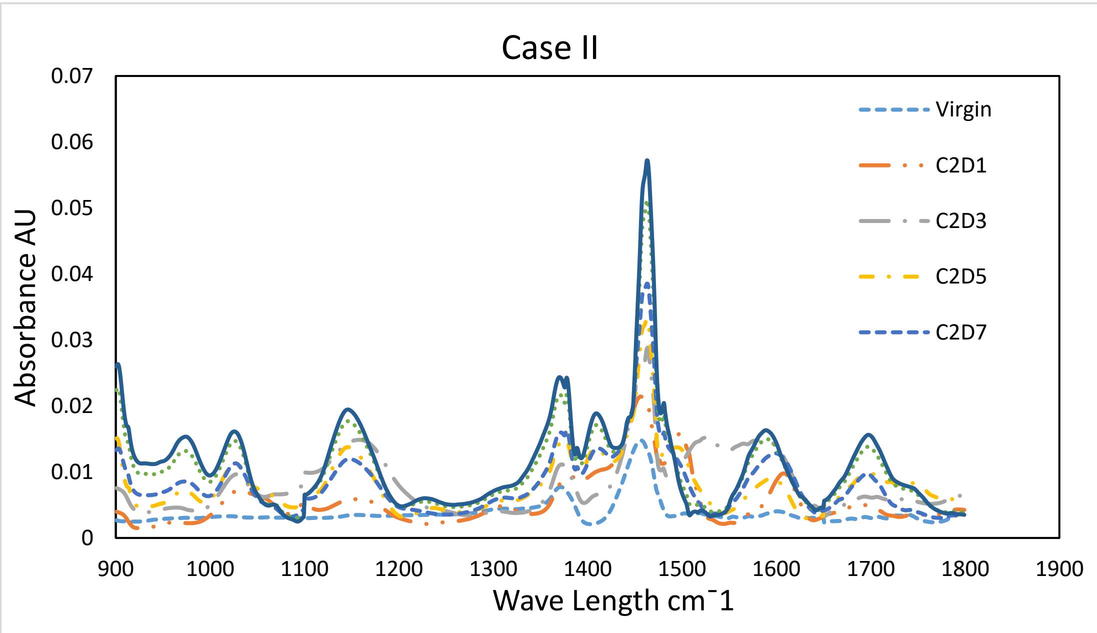


Figure 5.17 FT-IR spectrum of bitumen extracted from Case II samples

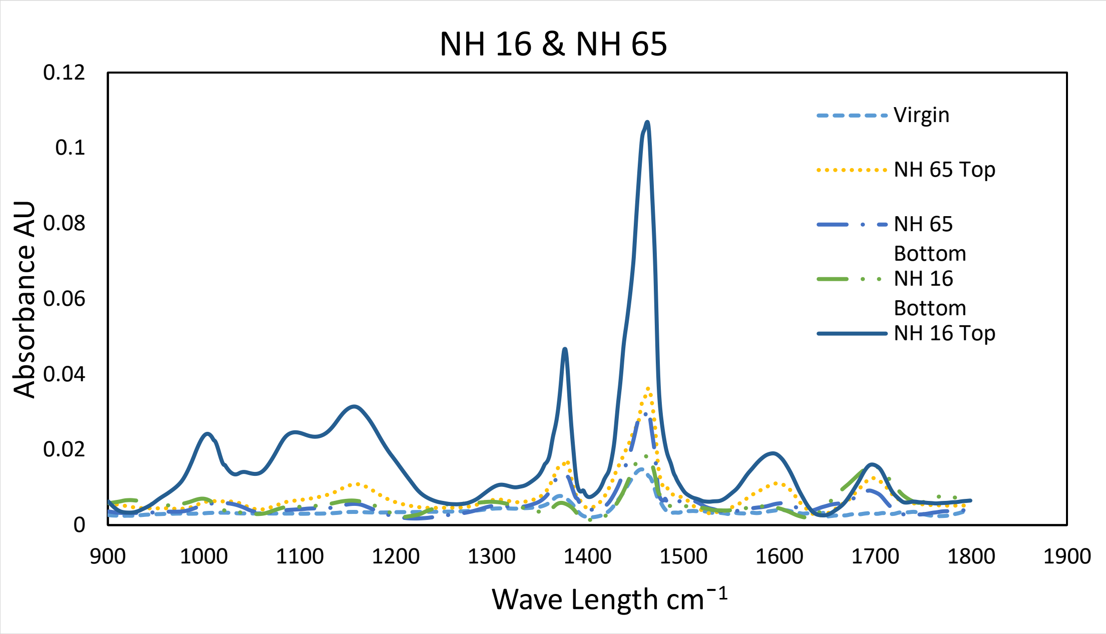


Figure 5.18 FT-IR spectrum of bitumen extracted from NH 16 and NH 65

Out of several methods available, two methods are widely used for quantifying the sulfoxides (S=O) and carbonyl compounds (C=O) at wave lengths 1030 cm $^{-1}$ and 1700 cm^{-1} respectively. The first method, known as area method, uses the ratios of area under the specific required peaks i.e., area under wave lengths 1030 cm $^{-1}$ (Sulfoxide index) or 1700 cm^{-1} (Carbonyl index) to the total area under the peaks. In the second method, known as amplitude method, the absorbance values at specific wavelengths (here 1030 cm $^{-1}$ and 1700 cm $^{-1}$) are divided with the absorbance values of the wavelengths that are not affected by oxidation. Generally, ethylene (CH2) and the methyl (CH3) groups are considered to be not affected by oxidation, therefore the absorbance at wavelengths 1375 cm $^{-1}$ and 1460 cm $^{-1}$ are taken as reference values. The equations 5.8 and 5.9 are used to compute Sulfoxide index and Carbonyl index.

The Sulfoxide index, $I_{SO} = \frac{\text{Absorbance value at 1030 cm} -1}{\text{Absorbance value at 1375 or 1460 cm} -1}$ --------- Eq. 5.8

Carbonyl index, $I_{CO} = \frac{\text{Absorbance value at 1700 cm} -1}{\text{Absorbance value at 1375 or 1460 cm} -1}$ --------------Eq. 5.9

The sulfoxides and the carbonyl indices for the bitumen extracted from Case I, Case II and National Highways, samples with respect to the aging periods are presented in the Table 5.14. It can be observed that the sulfoxide index of the bitumen extracted from the Case I samples showed a sharp increase after 5 days of aging. When Sasobit and hydrated lime are added to the mix, the sulfoxide index is almost constant without much variation as observed in Case II samples. With the progression of aging, the carbonyl index also increased. The variation of carbonyl and sulfoxide indices of bitumen extracted from the Case II samples is not much and this trend supports the fact that the Sasobit and hydrated lime are efficient in reducing the effects of oxidative aging in bituminous concrete mixtures. The data in Table 5.14 is graphically presented through Figures 5.19 and 5.20 for easy comparison between Case I and Case II samples.

Table 5.14 Sulfoxide and carbonyl indices of Case I, Case II and field Samples

	Aging Days	Sulfoxide Index	Carbonyl Index
Case 1	0	0.42587	0.55660
	1	1.06283	0.56373
	3	1.05234	0.56533
	5	1.23752	0.56082
	7	2.05022	0.65972
	10	3.07127	0.65972
	15	3.22142	0.68771
Case II	0	0.42587	0.55660
	1	0.33458	0.51582
	3	0.56642	0.52767
	5	0.47023	0.66941
	7	0.68675	0.60888
	10	0.74224	0.66410
	15	0.76777	0.67377
NH 65 Top	5 years	1.26754	0.62831
NH 65 Bottom	5 years	1.00867	0.53794
NH 16 Top	7 years	1.30036	0.66328
NH 16 Bottom	7 years	1.02586	0.54628

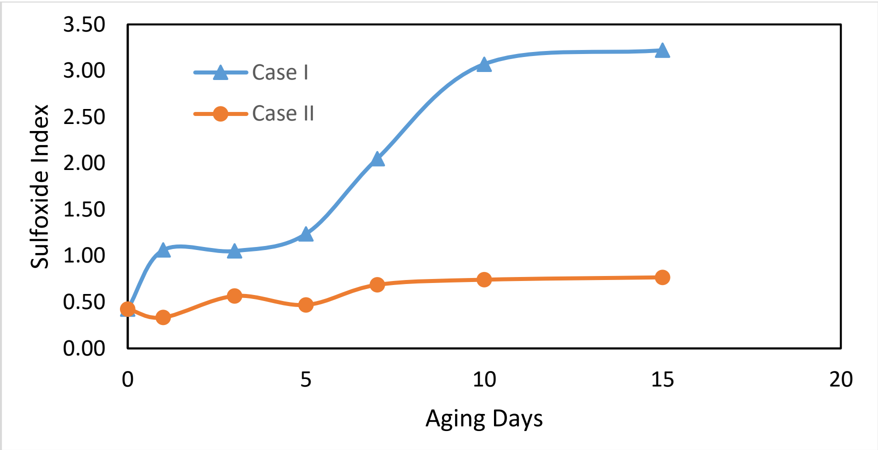


Figure 5.19 Post aged variation of Sulfoxide index for Case I and Case II samples

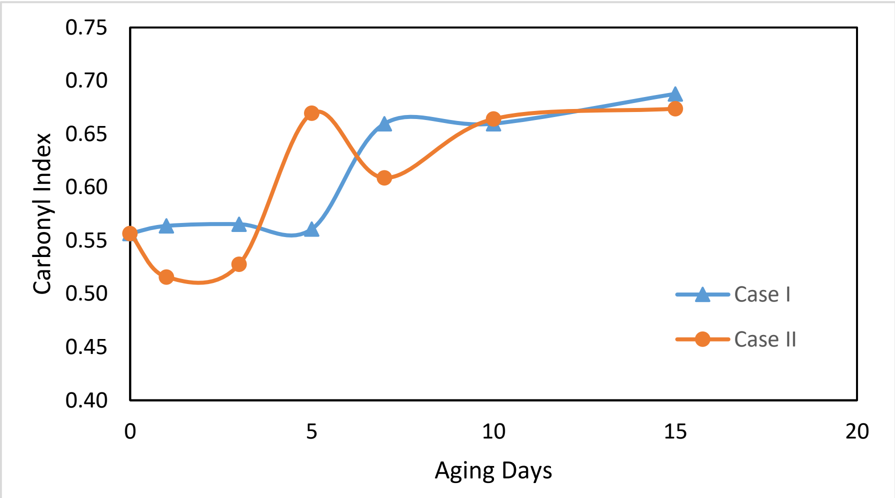


Figure 5.20 Post aged variation of Carbonyl index for Case I and Case II samples

The computed sulfoxide index values have shown an increasing trend when compared between 1-day and 15-day samples for both Case I and Case II. A sulfoxide index of 1.06284 was observed for 1-day aged Case I sample (without any modifiers), while the same index at the same age was 0.334586 for Case II samples (modified with hydrated lime and Sasobit). Similar trend can be seen for 3, 5, 7, 10 and 15 days aged samples. This clearly shows that the additives have demonstrated improved anti-aging characteristics.

The carbonyl index values have shown an increasing trend when compared between 1-day and 15-day samples for both Case I and Case II. A carbonyl index of 0.563733 was observed for 1-day aged Case I sample (without any modifiers), while the same index at the same age was 0.515827 for Case II samples (modified with hydrated lime and Sasobit). Similar trend can be seen for 3, 7, 10 and 15 days aged samples except for 5-day aged sample. It is interesting to note that the difference in carbonyl index values observed for Case I and Case II samples is not as prominent as seen for sulfoxide index.

It can also be observed that both carbonyl and sulfoxide indices have shown higher values for 7-year-old samples from NH 16, for top and bottom slices when compared with the respective samples collected from 5-year-old NH 65. This has strengthened the analogy that the older the sample gets; more will be the values of the above two indices.

A close examination of the sulfoxide indices observed for differently aged laboratory samples with those of field samples indicate that a 5-year field aging can be achieved with the corresponding oven aging of Case I samples for a period of approximately 5 days (122 hours as can be interpolated to achieve the comparative indices). Similarly, a 7-year field aging can be achieved with the corresponding oven aging of Case I samples for a period of approximately 5 days (124 hours).

5.3.5 SARA analysis on extracted bitumen from laboratory and field aged specimens

Bitumen is a material whose consistency gets influenced by the temperature related changes. The chemical characterization of the bitumen is usually done by saturates, aromatics, resins (together known as maltenes) and asphaltene (SARA) fractions.

The separated asphaltene and maltene fractions along with the hourly changes were found and presented, in terms of percentage of total bitumen taken, in Table 5.15. The maximum hourly rate of increase in asphaltene content (2.31% for Case I samples and 3.29% for Case II samples) was observed during the first day of exposure. The rate of change has considerably dropped afterwards, with a peak change being observed between 5 and 7 day exposed samples for both Case I (0.17%) and Case II (0.2%). The percentage asphaltene at the end of 15 days of exposure (51.33% for Case I and 46%

for Case II) clearly indicates that the Case I samples have aged more aggressively when compared with Case II samples. The asphaltene contents observed for field sample from NH 65 (43.17% and 39.50% for top and bottom slices respectively) was observed to be less than the corresponding samples from NH 16 (49.17% and 41.67% for top and bottom slices respectively), thus reconfirming that NH 16 (7 years aged) samples have aged more than the samples from NH 65 (5 years aged).

Table 5.15 Analysis of asphaltenes and maltenes

Aging	**Asphaltenes %**		**% increase / hour**		**Maltenes %**		**% decrease / hour**		**Maltene / Asphaltene Ratio**	
Hours	Case I	Case II	Case I	Case II	Case I	Case II	Case I	Case II	Case I	Case II
0	22.50	17.50	NA	NA	77.50	82.50	NA	NA	3.44	4.71
1 (24)	35.00	31.33	2.31	3.29	65.00	68.67	-0.67	-0.70	1.86	2.19
3 (72)	36.33	32.67	0.08	0.09	63.67	67.33	-0.04	-0.04	1.75	2.06
5 (120)	38.67	34.00	0.13	0.08	61.33	66.00	-0.08	-0.04	1.59	1.94
7 (168)	41.83	37.33	0.17	0.20	58.17	62.67	-0.11	-0.11	1.39	1.68
10 (240)	45.83	41.17	0.13	0.14	54.17	58.83	-0.10	-0.09	1.18	1.43
15 (360)	51.33	46.00	0.10	0.10	48.67	54.00	-0.08	-0.07	0.95	1.17
	Asphaltenes %		**Maltenes %**		**Maltene / Asphaltene ratio**		**Hours of aging required for Case I**		**Hours of aging required for Case II**	
NH 65 Top	43.17		56.83		1.32		200		300	
NH 65 Bottom	39.50		60.50		1.53		132		210	
NH 16 Top	49.17		50.83		1.03		324		Insufficient aging	
NH 16 Bottom	41.67		58.33		1.40		168		250	

For correlating the laboratory aging with natural aging process, maltene / asphaltene ratios for both laboratory and field aged samples were computed and presented in Table 5.15 and in Figure 5.21. Number of hours of laboratory exposure needed to make the aged sample equivalent with that of field samples is worked out by matching the corresponding maltene / asphaltene ratios of the laboratory aged samples, demonstrated in Figure 5.21. It can be clearly seen that more exposure was required to match the 7-year-old NH 16 samples than those from 5-year-old NH 65. Also, Case II samples have shown considerably better performance than Case I samples with increased exposure needed to match the field specimens.

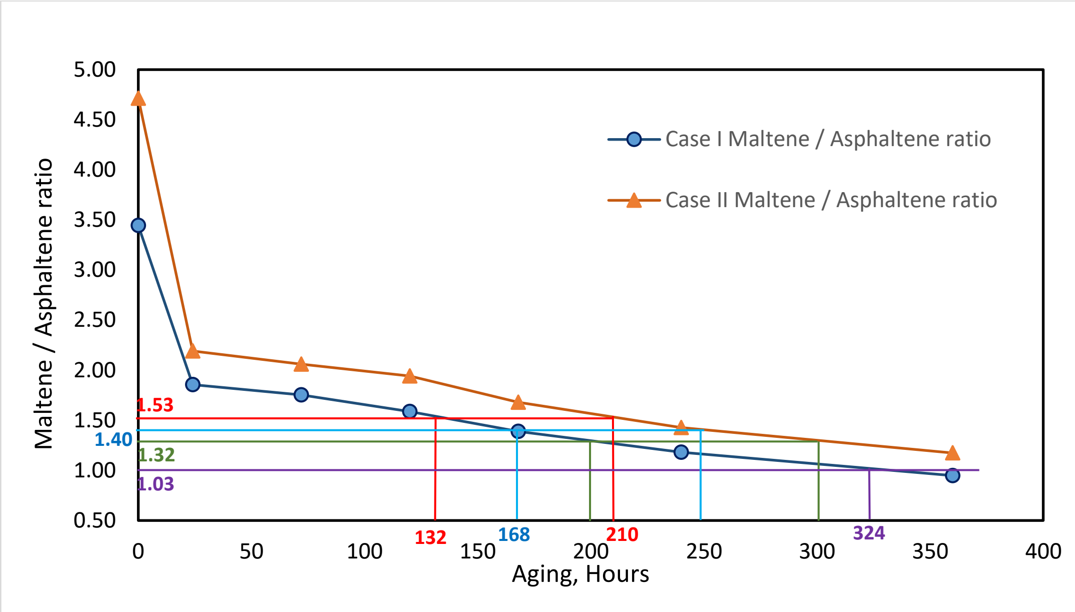


Figure 5.21. Variation of with maltenes / asphaltenes ratio with respect to aging

5.3.6 Morphological study results on extracted bitumen from laboratory and field aged specimens

FE SEM images of the bitumen extracted from unmodified Case I samples are shown in Figure 5.22. From the images presented in Figure 5.22, it can be clearly seen that the bitumen underwent a significant morphological transformation with aging. Prior to aging, the virgin bitumen consisted of a wavy pattern with many crests and troughs, typical with fresh bitumen samples. This pattern gradually got transformed with aging, as observed in the figure. Within one day, the patterns have shown clear change with shrunk wave forms. As the aging progressed in modified aging oven exposures, the morphology got transformed to crack like structure. Further, for the 7-day aged sample, almost smooth surface was observed. Particularly at 10 days of aging, the FE SEM images show a blank surface with crack like formations. However, at the end of 15 days of aging, the sample surface was observed to be missing peaks and valleys.

For the bitumen extracted from Case II specimens where optimized quantities of Sasobit Warm Mix Additive (WMA) and hydrated lime are added to the bitumen, the morphological changes in the samples, presented in Figure 5.23, are not as severe as those samples without these two additives (presented in Figure 5.22). The modified bitumen exhibited less wavy structure when compared with virgin bitumen. Similarly, the aged modified binders exhibited lesser severity when compared with the respective unmodified aged binders. This clearly proved the efficacy of the WMA and hydrated lime in reducing the tendency of aging.

The FE SEM images of bitumen extracted from NH 16 (Naturally aged by 7 years) and NH 65 (Naturally aged by 5 years) are presented in Figure 5.24. From this figure, it can be noted that the surface morphology of the extracted bitumen from the top portion is different when compared with the bitumen, extracted from the bottom portion of the core. Also, it can be observed that the samples from NH16 have shown more aging when compared with the corresponding samples from NH65, indicating large amount of aging occurred during the service period of the pavement. These images were found to be closely matching with the images obtained for lab aged samples corresponding to 10 and 15-day aging.

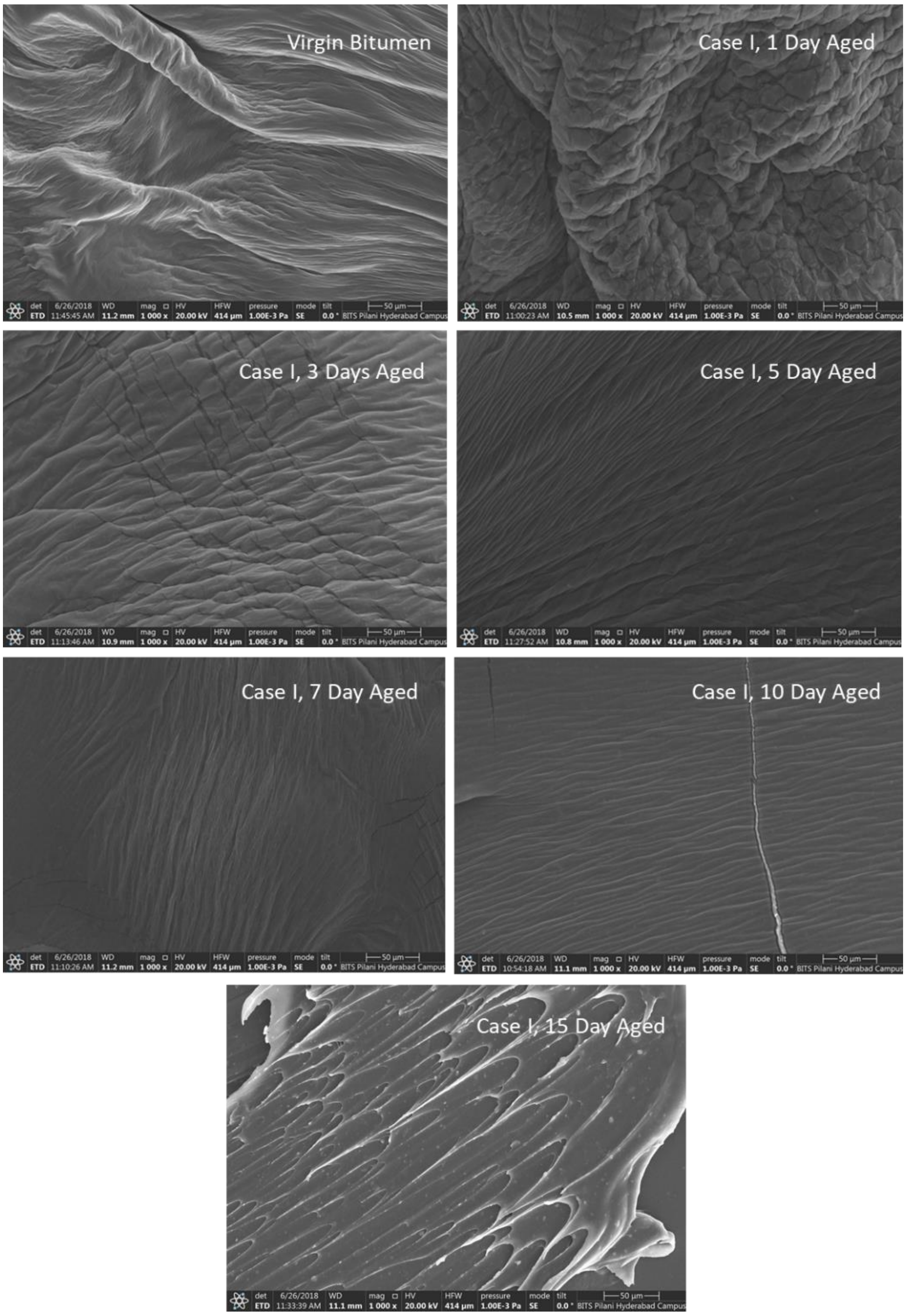


Figure 5.22. FE SEM images of virgin bitumen and the bitumen extracted from Case I samples

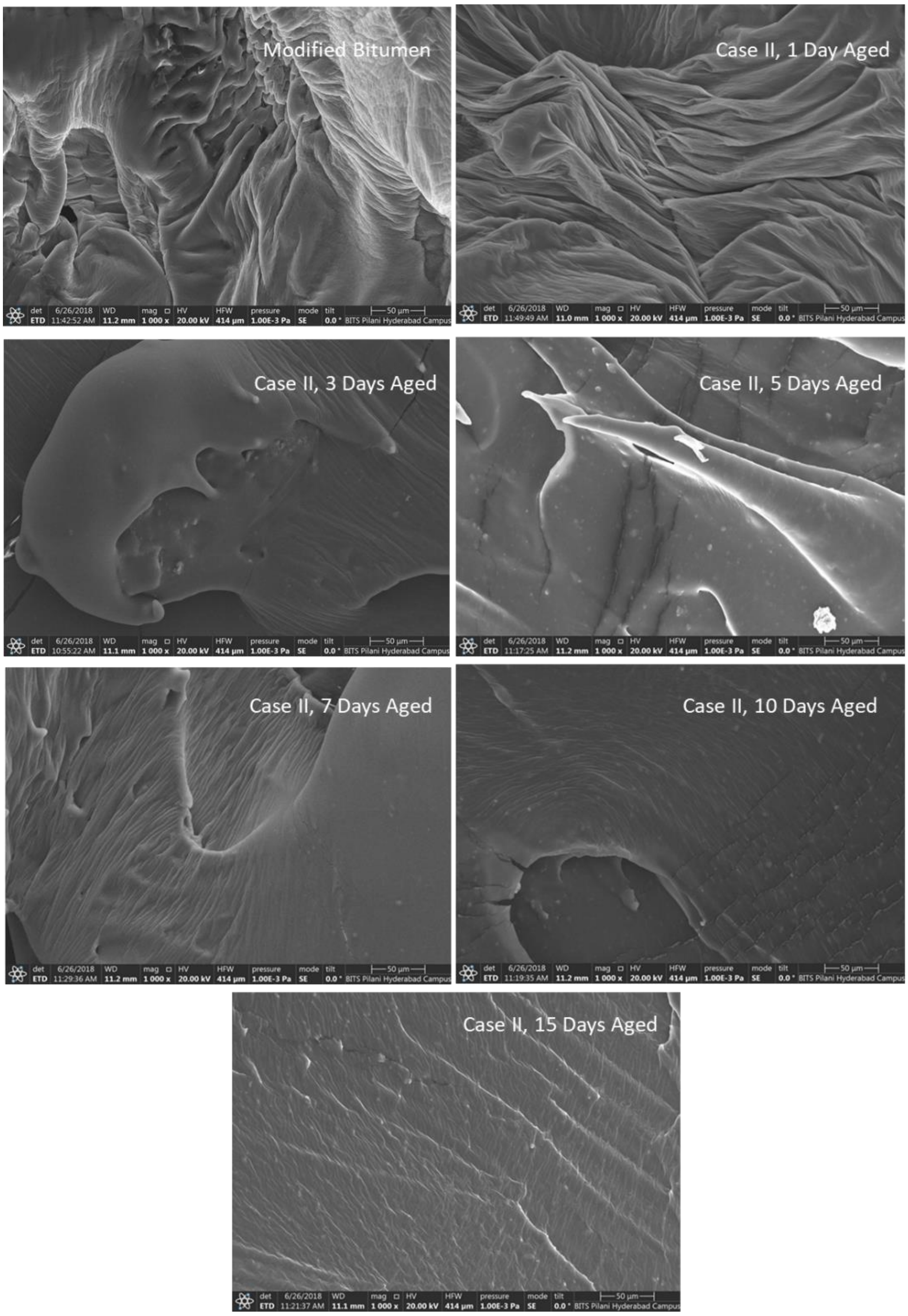


Figure 5.23. FE SEM images of Sasobit modified bitumen and the bitumen extracted from Case II samples

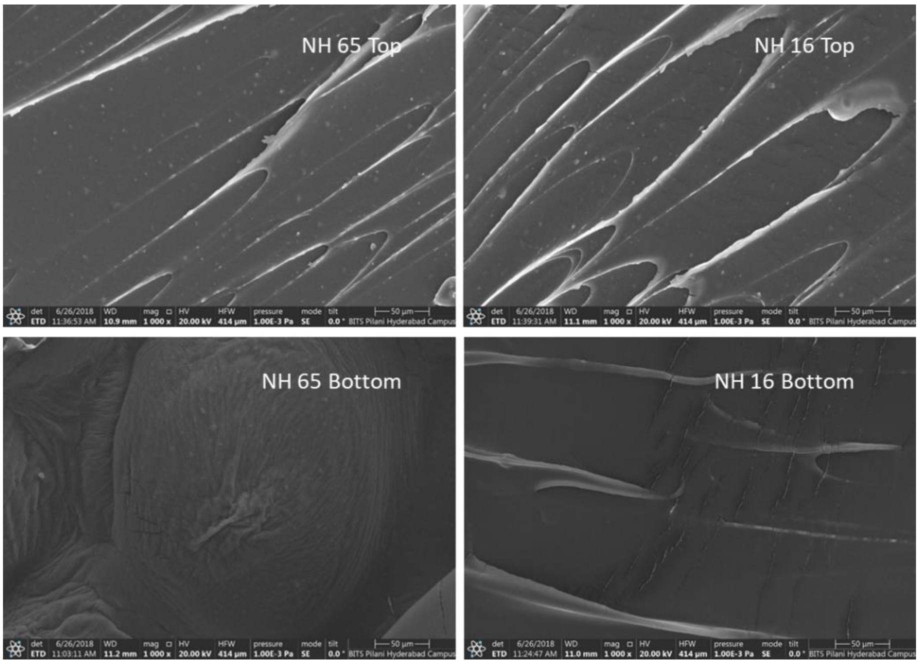


Figure 5.24. FE SEM images of the bitumen extracted from NH 65 and NH 16 cores

5.3.7 Energy dispersive X Ray (EDX) analysis on extracted bitumen from laboratory and field aged specimens

The variation of sulfur percentage obtained from EDX analysis, carried out on the bitumen extracted from Case I, II, NH 65 and NH 16 samples, is presented in Table 5.16. The data in Table 5.16 is graphically presented through Figure 5.25 for easy understanding. The percentage weights of the elements that are present in the samples are identified during this analysis. The increasing percentage of Sulfur with aging for both Case I and Case II samples clearly indicated that the oven exposure is resulting in aging process. The percentage weight of Sulfur for both NH 65 and NH 16 specimens is very high when compared with Case II samples under all the aging regimes, indicating that Case II samples are not aging to the extent of field even after 15 days of oven exposure. However, it can be observed that Case I samples needed close to 14 days of oven aging to simulate the aging occurred in 7 years old NH 16 field sample while 10 days aging of Case I samples was enough to simulate 5 years old naturally aged NH 65 sample.

Table 5.16 Energy dispersive X-Ray analysis (EDX) of extracted binder

	Aging Days	Sulfur (%)
Case I	0	5.32
	1	16.14
	3	16.81
	5	23.4
	7	38.46
	10	40.26
	15	48.57
Case II	0	5.3
	1	7.89
	3	10.14
	5	11.23
	7	13.49
	10	17.13
	15	24.35
NH 65 Top	5 Years	41.75
NH 65 Bottom	5 Years	10.13
NH 16 Top	7 Years	46.31
NH 16 Bottom	7 Years	11.67

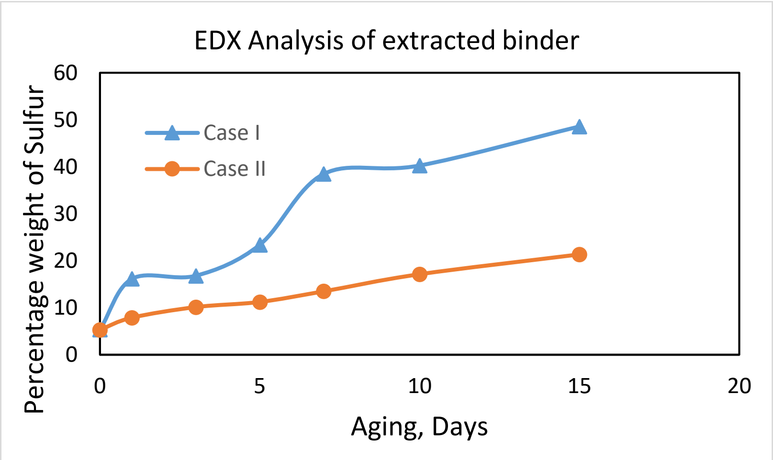


Figure 5.25. Post aged variation of percentage weight of sulfur for Case I and Case II samples

5.4 Statistical analysis of experimental results

The statistical analysis is conducted on results of the fifteen days aged bituminous concrete specimens and extracted bitumen. The experimental results are analyzed to find the mean, variance and weather the tests are significant. A “P Value two-tail test” assuming unequal variances, was conducted on the test results obtained from the physical, rheological, micro structural characteristics.

The analysis is carried according to following procedure:

Null hypothesis HO: The effect of hydrated lime and Sasobit on aging resistance of bituminous concrete specimens is same as the aging resistance of specimens without hydrated lime and Sasobit.

Alternate hypothesis HA: The effect of hydrated lime and Sasobit on aging resistance of bituminous concrete specimens is not equal to (more or less) of the aging resistance of specimens without hydrated lime and Sasobit.

We accept the null hypothesis (HO) if “P Value two-tail” is more than level of significance ($\alpha = 0.05$).

We reject the null hypothesis if “P Value two-tail” is smaller than level of significance ($\alpha = 0.05$) and conclude the alternate hypothesis (HA) is acceptable.

The results of the statistical analysis which was carried on the results obtained from the physical, rheological and microstructural investigations was presented through Tables Table 5.17 to Table 5.21 below.

Table 5.17 P-Value two-tail test results for Marshall stability, flow values and tensile strength ratio

	Marshall Stability		Flow Values		TSR	
	Case I	**Case II**	**Case I**	**Case II**	**Case I**	**Case II**
Mean	1610.06	1582.88	1.12	1.4	0.902	0.9275
Variance	2.873	4.597	0.017	0.005	7E-05	0.0003
Observations	5	5	5	5	5	5
Hypothesized Mean Difference	0		0		0	
df	8		6		4	
P(T<=t) one-tail	8.84E-09		0.0028		0.0232	
P(T<=t) two-tail	1.77E-08		0.0056		0.0464	

Table 5.18 P-Value two-tail test results for penetration, softening point and ductility tests

	Penetration		Softening Point		Ductility	
	Case I	**Case II**	**Case I**	**Case II**	**Case I**	**Case II**
Mean	13.5	14.1	87.48	86.2	11.96	16.46
Variance	0.165	0.55	0.577	0.615	0.523	1.553
Observations	5	5	5	5	5	5
Hypothesized Mean Difference	0		0		0	
df	6		8		6	
P(T<=t) one-tail	0.0218		0.0153		0.0002	
P(T<=t) two-tail	0.0437		0.0306		0.0004	

Table 5.19 P-Value two-tail test results for maltene/asphaltene ratio, sulfoxide index and carbonyl index values

	Maltene / Asphaltene Ratio		Sulfoxide Index		Carbonyl Index	
	Case I	**Case II**	**Case I**	**Case II**	**Case I**	**Case II**
Mean	0.948	1.166	3.225732	0.767318	0.68743	0.67335
Variance	0.0006	0.0013	0.0214	0.0001	0.0003	0.0004
Observations	5	5	5	5	5	5
Hypothesized Mean Difference	0		0		0	
df	7		4		5	
P(T<=t) one-tail	6.04E-06		1.53E-06		0.0767	
P(T<=t) two-tail	1.21E-05		3.05E-06		0.1535	

Table 5.20 P-Value two-tail test results for J_{nr} @ 0.1 kPa, J_{nr} @ 3.2 kPa and J_{nr_diff} (%) values

	J_{nr} @ 0.1 kPa		J_{nr} @ 3.2 kPa		J_{nr_diff} (%)	
	Case I	**Case II**	**Case I**	**Case II**	**Case I**	**Case II**
Mean	0.1714	0.1822	0.1928	0.2078	13.02	13.956
Variance	5.3E-06	1.87E-05	9.2E-06	2.67E-05	0.127	0.1230
Observations	5	5	5	5	5	5
Hypothesized Mean Difference	0		0		0	
df	6		6		8	
P(T<=t) one-tail	0.0013		0.0007		0.0015	
P(T<=t) two-tail	0.0026		0.0014		0.0031	

Table 5.21 P-Value two-tail test results for Sulfur (%) from, EDX analysis

	Sulfur (%)	
	Case I	**Case II**
Mean	48.51	24.354
Variance	0.662	1.8305
Observations	5	5
Hypothesized Mean Difference	0	
df	7	
P(T<=t) one-tail	2.36E-09	
P(T<=t) two-tail	4.72E-09	

From the above analysis, it can be observed that the results of the "P Value two-tail test" for all the investigations, which were conducted on the bituminous concrete specimens and extracted bitumen, were less than the level of significance ($\alpha = 0.05$) except for Carbonyl Index. So, the Null hypothesis is rejected, and alternate hypothesis is accepted. By observing the statistical analysis, it can be concluded that the combined effect of the hydrated lime and Sasobit is significant for all the physical, rheological, microstructural characteristics except for the Carbonyl Index. The failure of the Carbonyl Index as an aging parameter is already discussed in the section 5.3.4.

5.5 Summary of test results

With a view to provide the gist of what has been done in the present research work, a comprehensive summary of all the investigations is being presented below.

5.5.1 Summary of Marshall's stability and flow values of unaged and laboratory aged bituminous concrete specimens

- The aging has induced incremental stability with corresponding reduction in flow values for the bituminous concrete specimens. This phenomenon can be attributed to the fact that the aging induces additional stiffness with compromised elastic responses.
- It can be clearly seen that, during the initial stages of oven aging process, the change in Marshall's stability values were relatively higher for both Case I (without hydrated lime and Sasobit additive) and Case II (with hydrated lime and Sasobit additive) specimens respectively.
- With the advanced aging exposures, the corresponding changes in Marshall's stability values have become lesser, indicating that the rate of stiffening is falling with reduced influence of the aging at advanced exposure periods for both Case I and Case II specimens.
- It is interesting to note that the Case II specimens have resulted in comparatively more accelerated stiffening than the Case I specimens during the initial aging periods. However, at advanced aging periods, the Case I specimens stiffened more dynamically than Case II specimens.
- When Marshall's stability and flow values are observed, the Case I specimens have shown more aging tendency than the Case II specimens at the same exposure levels. This clearly has indicated that the hydrated lime and Sasobit (warm mix additive) have helped the mixture in reducing the aging tendency.

5.5.2 Summary of indirect tensile strength (ITS) and tensile strength ratio (TSR) of unaged and laboratory aged bituminous concrete specimens

- The Case II specimens outperformed the Case I specimens with increasing ITS (for both conditioned and un-conditioned specimens) and TSR values with progression of aging. This phenomenon can be attributed to the increased stiffness with aging process.
- It can be noted that the difference of conditioned ITS for Case II specimens is more prominent than for Case I specimens, indicating the improved performance against moisture attack of the Case II specimens due to the addition of hydrated lime.
- The rate of change of ITS values, kept on reducing for both conditioned and unconditioned specimens, with increase in aging exposure. This phenomenon can be attributed to the increased stiffening of bitumen during the initial aging exposures.
- The changes in TSR variation with time is better in Case II specimens due to hydrated lime.
- Case II specimens outperformed the Case I specimens with respect to conditioned and unconditioned ITS values, as well as TSR indicating the efficacy of hydrated lime and Sasobit additive.

5.5.3 Summary of fundamental properties of extracted bitumen from both field and laboratory aged samples

- The general tendency of the aged binders to show reduced penetration and ductility values with corresponding increase in softening point as well as penetration indices, is being observed.
- It can also be seen that the changes in bitumen properties are more prominent for Case I unmodified samples when compared with modified Case II samples, thus proving the efficacy of the modifiers in delaying the aging phenomenon. Interestingly, the changes were observed to be more prominent during the initial exposure.

- The effects of aging in terms of reduced penetration, increased softening points and reduced ductility values were more prominent in the Case of samples from NH 16, naturally aged for 7 years, when compared with the samples extracted from NH 65, naturally aged for 5 years.
- The physical properties of the extracted bitumen from bottom slices of both National highways indicate that the aging effects are not completely absent, though lesser in comparison with the corresponding top slices.
- The ductility and penetration values of extracted binder from laboratory aged samples and field core samples has indicated good correlation between the laboratory and field aging process.
- Softening point results of the field extracted bitumen from top slices are high when compared with the bitumen extracted from laboratory aged samples, indicating that the artificial aging, done in the modified oven as per AASTHO R30, is not sufficient to fully simulate the field aging.
- The top slice of National highways has aged more than the bottom slice by an equivalent of 1-day oven exposure in the laboratory. This reinforces the fact that the aging on the surface of the bituminous pavement will be more than the bottom layers.
- From the results of fundamental properties of extracted bitumen, it can be established that the laboratory aging with 5-day exposure, as recommended by AASHTO R30 guidelines, does not simulate the real field conditions fully.
- The Penetration retention percentage (PRP) values were gradually decreasing with incremental aging exposure. The PRP for the Case II binder was relatively higher at 79.30% after 1-day aging, when compared with the PRP of 73.35% observed for the unmodified Case I binder. As the aging progressed, the gap in PRP values of both modified and unmodified binders got considerably reduced (as observed in Table 5.9).
- The difference in aging of the bitumen extracted from the top and bottom slices in both the field Cases was observed to be same as that of the 1.5-day laboratory exposure of the Case I samples. Also, it can be noted that the 7-year-old NH 16 cores (both top and bottom slices) have aged more than the corresponding

samples of 5-year-old NH 65 cores by an equivalent half-a-day laboratory oven exposure of Case I samples.

- The Penetration Index (PI) was found to be increasing with increased aging exposure. The PI values of the bitumen extracted from the laboratory aged Case I samples are less when compared with PI values of the bitumen from Case II samples, thus proving the efficacy of the additives being tried in the current study in abating the aging process.
- The PI values of the top portion of the cores is more when compared with the bottom portions, indicating the rate of aging is more at the surface of the pavements when compared with the bottom portion.
- The Softening Point Increment (SPI) is another possible indicator for estimating the degree of aging that occurs in the bitumen binders. The SPI values for both Case I and Case II samples have shown growing trend with progressive aging in general.
- The SPI values of the top slices for both NH 65 and NH 16 indicated that the 15-day exposure may not be enough to simulate the field aging (top slice) as per this index. However, the SPI values of the bottom slices of both National Highways closely match with that of 9-day aged Case I samples.

5.5.4 Summary of Brookfield's rotational viscometer results

- The bitumen extracted from all the samples has shown Newtonian behavior, as expected from bitumen binders in the temperatures, ranging from 120 °C to 190 °C
- The addition of Sasobit and hydrated lime to bitumen has added value by reducing the viscosities marginally.
- The expected trend of enhancement in the viscosities with aging is clearly seen during the current experimentation.

5.5.5 Summary of Dynamic Shear Rheometer (DSR) analysis

- The complex modulus values of Case I samples are more than those of Case II samples for respective aging periods.
- It can be observed that the rate of aging that occurred in the Case II samples is uniform when compared with Case I samples. Also, it can be seen that the rate of aging between 10 and 15 days is more for Case I samples when compared with the Case II samples. These observations show that the addition of hydrated lime and Sasobit to the bituminous mixture has increased the anti-aging properties of the bituminous mixture.
- It was observed that level of aging that occurred in NH 65 (five years in-service) is almost equals to the 15-day aging simulated in laboratory but for NH 16 (Seven year in service), the level of aging that occurred in actual field conditions is more and as such, requires additional artificial aging and cannot be simulated using the recommended aging period of 5 days as suggested by AASHTO R30 guidelines.
- Based on **complex shear modulus aging index**, it can be observed that 14 days of oven aging was needed to simulate the 5 years of field aging, occurred in the top slice of NH 65. However, the corresponding bottom slices of the samples collected from the same site needed only 10 days of oven exposure. Similarly, the top slice of 7-year aged NH 16 samples could not be simulated fully even after 15 days of extended oven exposure while the bottom slices of the corresponding cores needed only 11 days of modified oven exposure for simulation. This clearly proves the fact that the top slice of 10 to 12 mm gets aged more dynamically than the bottom portion. Also, it is important to observe that the bottom portion too got aged to the extent that it needed 10 to 11 days of modified oven exposure.
- Based on **aging index developed from fatigue resistance parameter,** it can be deduced that the amount of aging that occurred in bitumen extracted from NH 65 bottom slice is equal to 11 days aging of Case I samples and 12 days aging of Case II samples. For the top slice of NH 65, aging occurred is around 12 days in Case I samples and 14 days for Case II samples. Similarly, for the bitumen extracted from NH 16 bottom slice, the level of aging is around 12 days

in Case I samples and 14 days for Case II samples. When it comes to the degree of aging in NH 16 top slice, the level of aging is equal to 13 days of Case I and 15 days for Case II samples.

- From the values of **aging index using loss tangent,** it can be noted that the aging index decrease with progression of aging periods for respective frequencies for both samples with and without additives and also from top to bottom slices.

5.5.6 Summary of Multiple Stress Creep Recovery (MSCR) analysis

- The J_{nr} and $J_{nr\text{-}diff}$ values, at both 0.1 kPa and 3.2 kPa loadings, are decreasing with respect to aging for both Case I and Case II samples.
- The values of J_{nr} were more for Case II samples for both loading conditions when compared with the Case I samples. This has clearly proved that the Case II samples, with Sasobit and hydrated lime additions, resisted the tendency of getting aged under similar conditions, when compared with Case I samples.
- The Jnr values of 7-year-old NH 16 are less than those of 5-year-old NH 65 indicating the excessive aging occurred in NH16.
- It can be observed that the J_{nr} values of the bitumen extracted from top portion of the pavements are less than those of the bitumen extracted from bottom portion, confirming that the top portion undergoes more aging in comparison with the bottom portion of the pavement.

5.5.7 Summary of Fourier Transform Infrared (FT-IR) Spectroscopy analysis

- Fourier Transform Infrared (FT-IR) Spectroscopy spectrums for Case I, Case II and field samples indicate that the 15-day laboratory modified oven aged sample has shown highest peak at the absorbance bands 1030 cm $^{-1}$ and 1700 cm $^{-1}$, when compared with other peaks with lesser aging exposure, thus proving the analogy that the peaks go up with aging.
- The absorbance values for Case II (with hydrated lime and Sasobit addition), having the similar aging exposure of 15 days, has shown lesser values when

compared with the corresponding sample belonging to Case I. This, again, proved the fact that the addition of hydrated lime and warm mix additive, Sasobit, in bitumen, had in fact, added value to bitumen in the form of improved aging resistance.

- The computed sulfoxide index and carbonyl index values have shown an increasing trend with aging for Case I and Case II samples. These values are more for Case I when compared with the Case II.
- The sulfoxide index of the bitumen extracted from the Case I samples showed a sharp increase after 5 days of aging. When Sasobit and hydrated lime are added to the mix, the sulfoxide index is almost constant without much variation.
- The variation of carbonyl and sulfoxide indices of bitumen extracted from the Case II samples is not much and this trend supports the fact that the Sasobit and hydrated lime are efficient in reducing the effects of oxidative aging in bituminous concrete mixtures.
- It is interesting to note that the difference in carbonyl index values, observed for Case I and Case II samples is not as prominent as that of sulfoxide index.
- The carbonyl and sulfoxide indices have shown higher values for 7-year old samples from NH 16, for top and bottom slices when compared with the samples collected from 5-year old NH 65. This has strengthened the analogy that the older the sample gets; more will be the values of the above two indices.

5.5.8 Summary of SARA analysis

- The maximum hourly rate of increase in asphaltene content was observed during the first day of exposure. The rate of change has considerably dropped afterwards, with a peak change being observed between 5-day and 7-day exposed samples for both Case I (0.17%) and Case II (0.2%).
- The higher percentage asphaltenes observed in aged Case I samples, when compared with aged Case II samples, proved that the addition of WMA and hydrated lime, has resulted in relatively lesser long term aging, thus clearly

indicating the influence of these additions in decelerating the aging phenomenon.

- The higher asphaltene content, observed for field samples collected from 7-year old NH 16, when compared with 5-year old samples collected from NH 65, confirmed that the surface course of NH 16 has undergone more aging than NH 65 surface course.

5.5.9 Summary of Field Emission Scanning Electron Microscope (FE SEM) Analysis

- FE SEM images of the bitumen samples extracted from Case I specimens shows that the bitumen underwent a significant morphological transformation with aging. The surface morphology of the samples from NH 16 has clearly indicated more aging when compared with the respective samples from NH 65.
- For the bitumen extracted from Case II specimens, the morphological changes in the samples were not as severe as those samples without these two additives. The aged modified binders exhibited lesser severity when compared with the respective unmodified aged binders.

5.5.10 Summary of Energy dispersive X Ray (EDX) analysis

- The increasing percentage of Sulfur with aging for both Case I and Case II samples has clearly indicated that the oven exposure is resulting in progressive aging process.
- The percentage of Sulfur is more for bitumen extracted from Case I samples when compared with the Case II samples, indicating that the level of aging occurred in the unmodified Case I samples is higher than that occurred in Case II modified samples.

Total modified oven exposure, needed for Case I specimens, to match the performance of the bitumen extracted from field cores, with respect to multiple parameters tried during the current research, are summarized at one place and presented through Table 5.22 for reference.

Table 5.22 Number of days required for Case I specimens in the modified oven to match with field aging based on the multiple criteria

	Exposure (in days) required for Case I samples in the modified oven to simulate field condition matching cores from ↓			
Property	**NH 65 Top**	**NH 65 Bottom**	**NH 16 Top**	**NH 16 Bottom**
Ductility	14	13	15	14
Penetration	14	13	15	14
Softening point	**	8	**	8
Penetration retention percentage	14.5	13	15	13.5
Complex shear modulus based aging index	14	10	**	11
Fatigue resistance parameter	12	11	13	12
FT-IR Sulfoxide Index	5	3	5.5	3
Maltene / Asphaltene ratio	8.3	5.5	13.5	7
EDX	10	1	14	1
**** Modified oven aging was not enough to simulate**				

It can be observed from Table 5.22 that there is a need to further investigate and standardize the criteria to be considered for understanding the exact exposure needed to simulate the field conditions.

CHAPTER 6

CONCLUSIONS

6.1 Introduction

During the current research, major target was to simulate the field aging process with laboratory aging, as closely as possible. It was in this direction that the 5-year and 7-year field aged specimens were collected by coring at two different field sites. The bitumen residue, extracted from these field cores, through Sohxlet extractor, was subjected to all the necessary tests including micro structural and rheological investigations.

Bituminous concrete specimens were prepared with similar specifications as that of field cores. To fulfill the primary objective of comparing the natural aging with accelerated lab-based aging of bituminous concrete mix, it was decided to follow the AASHTO R30 guidelines, with a few necessary modifications, to take the research closer towards achieving the intended objective. Further, the extracted bitumen samples were investigated thoroughly for understanding the transformations taking place with lab-based aging. A comparative assessment was carried out to judge the closeness with which the lab-based aging could be matched with the field aging process.

The influence of warm mix additive i.e., Sasobit and hydrated lime on aging process was also investigated by making adequate number of specimens (Case II) and subjecting them to the same aging regimes, which were attempted for the Case I specimens without hydrated lime and Sasobit. The bitumen extracted from field aged samples was also tested and the performance was compared with that of the lab aged samples for assessing the level of match between the laboratory and field aging processes. The conclusions arrived are listed below.

6.2 Conclusions

The aging phenomenon of bituminous binder is a very complex process, involving numerous factors affecting it. The current research focused on the performance aspects of the bitumen and bituminous concrete subjected to aging and the conclusions are presented below.

- The conclusions, from laboratory aged bituminous concrete specimens:

    - The flow values of Case I specimens have reduced from 3.2 mm for unaged specimen to 1.3 mm for 15-day aged specimens. Similarly, the flow values for Case II specimens have reduced from 3.8 mm to 1.6 mm for the same aging regime. This shows that the workability of the mix with hydrated lime and Sasobit is better than the workability of the mix without hydrated lime and Sasobit.
    - The percentage increase in Marshall's stability values were relatively higher (8.44% and 11.32% for Case I and Case II specimens respectively) after first day of aging exposure. This is because of addition of hydrated lime.
    - Indirect tensile strength (dry) values of Case I specimens have increased from 0.61 MPa (at zero aging exposure) to 0.91 MPa after 15days of laboratory aging exposure. Similarly, Indirect Tensile Strength values (dry) of case II specimens have increased from 0.67 MPa (at zero aging exposure) to 0.93 MPa after 15 days of laboratory aging exposure. The increase in stiffness value of Case I aged specimens is around 50% with respect to unaged specimens and 40% for Case II specimens. This shows that there is significant reduction in aging in Case II samples.
    - The tensile strength ratio (TSR) values of Case I specimens increased from 0.77 at unaged condition to 0.90 after 15-days aging. The corresponding values for Case II specimens increased from 0.82 to 0.92 during the same aging exposure. This shows that the moisture damage is significantly lower for hydrated lime and Sasobit added (Case II) specimens due to anti-stripping properties of hydrated lime.

- The rate of change of ITS values was observed to be gradually reducing for both conditioned and unconditioned samples with advanced aging exposure. This phenomenon can be attributed to the tendency of bitumen getting stiffened rapidly during the early aging exposure periods when compared with what happens during the later stages of aging exposures. The changing asphaltene, maltene ratios with aging play a critical role in this phenomenon.

- The conclusions from the properties of extracted binder (laboratory and field samples):

  - The rate of change of penetration, softening points and ductility values for Case I aged bitumen is considerably higher when compared to Case II aged bitumen samples. This clearly indicates the efficacy of adding the Sasobit (warm mix additive) and hydrated lime to the mixture.
  - With the aging, both ductility and penetration values of extracted binder from laboratory aged and field core samples has indicated good correlation. Softening point results of the field extracted bitumen from top slices are high when compared with the bitumen extracted from laboratory aged samples, indicating that further oven aging with conditions like UV exposure, may be necessary to make the laboratory and field aged specimens comparable with regard to these properties.
  - The bitumen extracted from the top 12 mm slices of the cores collected from the field got aged more aggressively than the bitumen extracted from the bottom portion of the same cores, with regard to the changes in the values of penetration, softening point, penetration Index and ductility, with aging. This phenomenon can be attributed to abundant availability of oxygen along with direct exposure to the ultraviolet and other solar sourced radiations at the top, when compared to the bottom portion of bituminous concrete layer.
  - The complex shear modulus values of bitumen extracted from NH 65 bottom slices are almost equal to the values of 10-days aged Case I and Case II samples. Similarly, the complex shear modulus values of NH 65 top slices are almost equal to 15-days aged Case I samples and more than 15 days aged

Case II samples. This shows the aging is significantly reduced by addition of hydrated lime and Sasobit.

- The complex shear modulus values of NH 16 bottom slices are in between 10-day and 15-day aged Case I and Case II samples. The values of NH 16 top slices are almost equal to the complex shear modulus values corresponding to 15-day aged Case I and Case II samples.
- Penetration index, softening point increment, complex shear modulus-based indices, fatigue resistance parameter, loss tangent are being used to assess and establish the level of aging for both laboratory and field aged samples. These indices provided a logical base to compare both the aging processes. In MSCR test, when the J_{nr} values of the samples from NH 65 and NH 16, were compared with the Case I samples, it can be noted that around 14 days of laboratory oven aging was necessary to simulate the aging occurred in 5 year old top slices of NH 65. It can also be noted that the artificial laboratory oven aging is not enough to simulate the actual aging that occurred in the 7-year-old top slices of NH 16.
- Sulfoxide index obtained from FT-IR analysis has indicated that the fresh sample, when aged for 122 hours (approximately 5 days), has matched the corresponding index found for the sample extracted from top slice of 5-year-old field core. This shows that the Sulfoxide index is very sensitive when compared with the other aging indices used as number of days laboratory simulation required are very less.
- Though the sulfoxide index has helped in matching with the field aged samples, complete match could not be achieved with respect to other carbonyl indices of the extracted binders.
- Maltene to asphaltene ratio can be used as a good indicator to understand the aging of bitumen samples. Using this property, while 8.3 days of lab aging was required for Case I samples to simulate the filed aging of 5 years, it needed 12.5 days of similar aging for Case II samples. Same trend is being observed for the samples from 7-year-old NH16 also. The conversion of maltenes to asphaltenes is significantly reduced by the addition of hydrated lime and Sasobit.

- The percentage of Sulfur obtained from EDX indicated that the Case I samples underwent more aging when compared with the Case II samples. It was also established that the top portion of the National highways underwent more aging than the bottom portion. FE SEM images also indicate the same.

6.3 Research contributions

The modified oven aging, rigorous testing and analysis of the bituminous concrete specimens (with and without hydrated lime and Sasobit) and bitumen extracted from the aged specimens, resulted in the following specific contributions:

- The modified oven aging procedure is developed, as a part of the current research, making changes to the existing AASTHO R30 guidelines. This effort has improved the efficiency in aging the specimen considerably when compared with the existing guidelines.
- The modification suggested to the AASHTO R30 guidelines, with regard to the controlled supply of oxygen, is simple and can be implemented without much difficulty in any laboratory.
- The correlation between the laboratory aging through modified oven and the actual field aging was established with various tests (fundamental properties, rheological properties, and microstructural analysis) on extracted bitumen from the laboratory samples and field samples. These correlations can aid the researchers to progress further, when attempting to understand the way the bitumen ages in the field.
- The efficacy of the hydrated lime and warm mix additive (Sasobit) in reducing the effects of aging that occurs in bitumen binder was thoroughly established through rigorous testing.
- Both sulfoxide index and carbonyl index were being thought to be representing the aging phenomenon of bitumen. However, it was clearly established that only sulfoxide index is consistent in establishing the aging aspect while the carbonyl index did not help much in understanding the level of aging fully.

- The suggested Maltene to Asphaltene ratio obtained from SARA analysis is found to be a good indicator for understanding the aging of bituminous binder and can be used for correlating the accelerated laboratory aging with that of field aging.

6.4 Limitations and future scope of the work

- The amount of laboratory aging induced on the specimens is suitable to simulate the existing environmental variables prevalent at the sites from where the core specimens were extracted. When a similar study is carried out at other locations with different environmental variables, the exposure periods and other conditions may need revision.
- The combined effect of UV rays along with oxygen supply was not attempted during the current research. Hence, this aspect can be taken up as future scope of research.
- The current study was limited to virgin VG 30 grade of bitumen alone. Similar studies can be carried out for other modified and unmodified grades of bitumen binders.
- The present study is carried out for MoRT&H's Grade I mixture alone. Similar studies can be carried out for all the other surface course options available in use.
- Mapping the extent of aging in recycled asphalt pavement (RAP) material with the corresponding performance of RAP based mixture would provide a true insight in this area of research.
- During the current study, the combined influence of hydrated lime and warm mix additive (Sasobit) was studied. The study can be extended by investigating the individual influence of hydrated lime and warm mix additive.
- Inter relationships between consistency tests, rheological investigations and microstructural investigations can be established with view to explain the future behavior in a better manner.

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As a researcher, he also published his works in many journals of international repute and presented his works in different national and international conferences. He has a multiple textbook chapters published to his credit and also been a co-author for an NCERT book on Transportation Engineering for 11th grade students in India. He has guided two scholars for their Ph.D. s at BITS Pilani Hyderabad Campus and currently guiding two scholars and co-guiding two more scholars as a part of inter disciplinary research activity. He has close to three decades of academic experience having guided number of masters and bachelor’s thesis works. He has been an active consultant for design and investigations of Runways, Expressways, National Highways as well as Village Roads.

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He is one of the two researchers who was entrusted with the task of revising the IRC:SP: 98-2020 " Guidelines for the use of waste plastic in hot bituminous mixes in wearing courses" and IRC: 129-2019 "Specifications for open-graded friction course". His works were published in many journals of international repute he delivered lectures in numerous educational institutes and organizations as a guest and keynote speaker. He has a patent granted for "A process for the preparation of waste plastic modified bitumen useful for rut resistant and water-resistant bituminous mix for road construction"- Patent Number 246060, Indian Patent Application Number-2309/DEL/2004. He has applied for a patent on the "Process for the Development of a Bio-based Encapsulation for Bitumen Pellets for Cold Supply", December 2019. He is currently guiding three scholars for their Ph.D. and co-guiding two more scholars at BITS Pilani Hyderabad Campus. He is the Principal investigator in three ongoing research grants one of which is "Mitigation strategy to counter top-down cracking due to non-uniform contact stresses in Flexible pavements" sponsored by NHAI.

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