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CHARACTERISATION OF BITUMEN AND ASPHALT MIXTURE WITH RECYCLED WASTE PLASTIC (RWP) MODIFIED BINDER

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ABSTRACT

Good bitumen properties are desirable nowadays to achieve longer service life. In order to enhance the quality of bitumen in term of its properties, many researchers began to focus on bitumen modifications particularly in modifying bitumen with polymer mainly from recycled waste plastic. Incorporating bitumen with polymer significantly improves and reduces asphalt pavement distresses. However, there are still some drawbacks which significantly limit its development namely inappropriate polymer and bitumen blend may result in poor storage stability that would affect the performance of asphalt mixture. Besides that, excessive hardening by adding more polymer in bitumen may increase the energy required during production which will increase the cost of the construction. Also, the modification procedure may need high shear and mixing temperature for some period that would change the rheological properties of Polymer Modified Bitumen (PMB) due to ageing. Moreover, the increasing amount of plastic waste material at the landfill is another vital concern. So, the main aim of this study is to re-use the recycled waste plastics as a modifier in bitumen to enhance the performance of asphalt mixture. Therefore, finding the optimal shear rate, modification temperature and the mixing time are essential to ensure the optimum blend of Recycled Waste Plastic (RWP) modified binder that is stable and better in performance could be produced. In this study, six types of recycled polymer and bitumen with penetration grade of 160/220 are used for the modification. Then, additional two binders namely control binder (penetration grade of 40/60) and SBS-modified bitumen were used to compare with the RWP-modified binders that gave significant improvement in terms of its performance.

There are three main phases involved in this study. It begins with Phase 1 (selection and incorporation of RWP in bitumen) for optimising the blending condition for each RWP, Phase 2 (binder test) is to identify the rheological properties of PMB and optimise the RWP concentration and lastly, Phase 3 (mixture performance test) is to determine the performance characteristics of asphalt mixture for each RWP with respect to fatigue cracking and rutting.

Phase 1: The potential of RWP to modify base binder were identified based on the melting point of RWP and the optimisation of the blending variables were done based on the softening point of the modified binders. Then, RWP that cannot be blended with base binder will be excluded from the testing programme.

Phase 2: At this phase, the binder tests for RWP-modified binders in terms of chemical, structural, rheological, performance and durability properties were carried out. The effect of ageing during blending of RWP-modified binder was analysed by means of Fourier Transform Infrared (FTIR) for the chemical approach. The structural property was assessed in term of the stability of RWP-modified binder at higher temperature storage. Then, the rheological characteristics for unaged and aged binders were investigated. The fatigue and rutting performance of binder were characterised. The fatigue testing was evaluated by means of the Linear Amplitude Sweep (LAS) test and the Double-Edged Notched Tension (DENT) test while the rutting testing involved the Multiple Stress and Creep Recovery (MSCR) test. Lastly, the durability of the binders against moisture damage was investigated using the Peel test.

Phase 3: This phase consists of performance of asphalt mixture. First, an aggregate of granite and typical dense gradation of 14mm was selected for designing all asphalt mixtures by using two binder contents. The stiffness, fatigue and rutting were evaluated and conducted using Indirect Tensile Stiffness Modulus (ITSM) test, Indirect Tensile Fatigue Test (ITFT) and Repeated Load Axial Test (RLAT). All these tests were carried out by means of the Nottingham Asphalt Tester (NAT) machine.

The result from the studies show that the RWP-modified binder can significantly improve the rheological properties and performance of the base binder. The improvement is highly dependent on the amount of RWP added and more pronounced at higher percentage of RWP added namely 7%. However, the RWP-modified binders are more susceptible to fracture but it could withstand higher strain levels due to higher viscosity values that make it stiffer. The improvement in moisture durability for all binders after conditioning show that the effect of stiffening is dominated in which ageing take place during the conditioning The result obtained from the asphalt mixture performance test show that the RWP-modified mixtures are better than conventional mixture in rutting performance but not in fatigue performance.

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DECLARATION

The research described in this thesis was conducted at the Nottingham Transportation Engineering Centre, Civil Engineering Department, the University of Nottingham between February 2015 and September 2019. I declare that the work is my own and has not been submitted for a degree from another university.

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CHAPTER 1: **INTRODUCTION**

1.1 Background

Bitumen is a sticky, black or dark brown and naturally viscous liquid or semi solid in some deposits at room temperature (McNally 2011). It is the residue of fractional distillation of crude petroleum which is formed by nature that occurs over millions of years and the heavy end product in the petroleum refinement. In the building and engineering materials applications especially in pavement industry, bitumen plays vital role of low-cost thermoplastics as a binder in flexible pavement. In fact, the good quality of bitumen is indispensable for earning the maximum economic benefits parallel with increase road traffic volume and vehicle loading. Zhu et al. (2014) reported that some good bitumen properties can be obtained by selecting good crude oil or proper refinery process but limited oil resources for producing and controlling the quality of bitumen is inevitable. Good adhesion and cohesive binding property and able to resist ageing are desirable.

In order to enhance the quality of bitumen in term of its properties, many researchers began to focus on bitumen modifications particularly in modifying bitumen with polymer (Collins et al. 1991; Ait-Kadi et al. 1996; Cardone et al. 2014; Yu et al. 2015). Bitumen modification will produce the pavement which is more resistant to fatigue, thermal cracking, rutting, stripping, and temperature susceptibility. Specifically, significantly produce better improvements of road pavement properties during in service when subjected to adverse weather conditions and minimize the effects of increasing traffic loads are the main principle of adding polymer in bitumen (Ezzat 2012; Köfteci et al. 2014).

Synthetic and virgin polymers have been used in bitumen as a modifier as early as 1843 (Kalantar et al. 2012; Yu et al. 2015). Unfortunately, virgin polymers are uneconomical to use because these materials are expensive which cause only a small amount can be used to enhance the bitumen properties. Currently, the waste polymer has come into consideration for modifying bitumen due to the high cost of virgin polymer. Numerous studies have shown that the modification of bitumen with recycled polymers show analogous results in enhancing the properties of bitumen as compared to virgin polymers (Murphy et al. 2000; Garcıa-Morales et al. 2004; Garcia-Morales et al. 2006; Fang et al. 2009; Kalantar et al. 2010; Kumar & Garg 2011; Ezzat 2012; Behl et al. 2014; Köfteci et al. 2014). The addition of recycled polymer significantly improves various bitumen properties. Besides that, it was observed that those bitumen properties yield substantial improvement in the performance and quality of asphalt mixture to be more stable and harder at high temperatures and more flexible at low temperatures (Ismail et al. 2011; McNally 2011).

At the landfill, plastic waste materials do not decay and will remain in nature for many years. This will cause pollution to the environment. To solve this problem, many companies start to produce recycled plastic from the plastic waste, resulting in decreasing amount of plastic waste to be burnt underground. From economic and environmental points, besides enhancing the performance of bitumen, reutilisation of recycled plastic in bitumen may contribute to solve the waste disposal problem (Kumar & Garg 2011; Kalantar et al. 2012; Nassar et al. 2012; Modarres & Hamedi

2014) in which tonnes of waste plastics are produced every year and require more places at landfill.

Despite the advantages of polymer modification have been reported, there are still various drawbacks occurs which could limit its future development (Zhu et al. 2014). Polymers as modifier could create difficulties in dispersion in bitumen, phase separation problems, poor storage stability, higher viscosity at layout temperature, low thermal fatigue cracking resistance, low resistance to heat and oxidation and high cost (Read & Whiteoak 2003; Kalantar et al. 2012).

1.2 Problem Statement

In the road paving application, bitumen experiences a numerous thermo-mechanical states during its service life. In general, at low temperature or/and high rapid loads, bitumen behaves like elastic solid. When applying too many loads to the pavement, the behaviour of bitumen which is elastic solid may break and eventually would become brittle and crack will occur if its load limit is exceeded. These problems will lead to the crack initiation and propagation. While at high temperature service and/or slow moving loads, the bitumen is viscous (liquids) and can flow. Pavement deteriorations occur attributed of multiple factors namely traffic loads, construction practice and thermal susceptibility of asphalt pavement (Ait-Kadi et al. 1996; Pérez-Lepe et al. 2003; Cortizo et al. 2004; Al-Hadidy & Tan 2008).

The conventional road pavement easily deteriorates. Early in pavement life rutting is the main concern, while later in pavement life fatigue cracking becomes the major concern (Behl et al. 2014). Besides that, bitumen by themselves will not

significantly improve performance of the binder. Therefore, the idea of adding polymer by means of binder modification to enhance the performance of bitumen at all in service temperature range is adopted. Polymer modified bitumen (PMB) need to be stiff and elastic to withstand traffics loads at high temperature to prevent rutting or permanent deformation while at low temperature; it should be flexible, lower viscosity and good ageing resistance in order to prevent fatigue and thermal cracking. Ait-Kadi et al. (1996) also stated that it needs to withstand the thermal cycling of service without cracking or deforming.

However, there are still various deficiencies appeared when modifying bitumen with polymer which may cause limiting its performance. These deficiencies are:

1. The modification of bitumen with polymers requires a better understanding of the interrelationship of the bitumen and polymer which is on the other hand is the reaction of polymer modification mechanism (McNally 2011; Fang et al. 2014b). Inappropriate polymer and bitumen blend may result poor storage stability of the modified bitumen (Pérez-Lepe et al. 2006), disturb the dynamic equilibrium and reduce the homogeneity of the bitumen (Fang et al. 2014b), thus would affect the performance of pavement. Many studied have been conducted for enhancing the compatibility and stability of PMB by adding reactive polymers (Nassar et al. 2012), sulphur vulcanization (addition of antioxidants), functionalization (adding functional group), and adding hydrophobic clay minerals (Zhu et al. 2014) but the exact reaction mechanism is still remain unclear and involve high cost. Polacco et al. (2005) reported that

no sufficient enhancement on compatibility was achieved when adding compatibilising group.

2. The use of polymers may result in stiffer and harder HMAs which is going to decrease the workability, cause difficulties in transporting, placement and compaction. This would lead to the increase in energy required during mixing and compressing because of excessive hardening (Köfteci et al. 2014). It should not be too viscous at mixing temperature or too brittle at low temperature to improve its efficiency (Modarres & Hamedi 2014). So, the amount of polymer to be used in the blend should be adjusted for getting a proper value of viscosity at the temperatures involved in bitumen application and compaction (Garcia-Morales et al. 2006).
3. The modification procedures may have significant effect on the properties of the resulting blend. In order to obtain a homogeneous blend of polymer and bitumen, high shear and mixing temperature are required. Consequently, the process of ageing has occurred due to the oxidation of bitumen and/or degradation of polymer that will change the rheological properties of PMB (Lu & Isacsson 2000; Airey 2003). Nevertheless, some polymer do not require high shear rate mixer to well disperse in the bitumen although the time of blending can be reduced when using high speed (Al-Dubabe et al. 1998) and blending PMB under low shear mixing for a few hours could allow the polymer to swell in to the bitumen and produce a homogeneous mixture (Hunter et al. 2015).
4. Polymers have the advantage of utilizing the desirable properties of different functional groups in the same molecule and have the possibility to help enhancing aggregates adhesion. However, none of them were specially

designed for enhancing adhesion and their capability to help anti-stripping is quite limited (Zhu et al. 2014) may affect the usage of PMB in improving the durability performance of road pavements.

5. There are a wide range of plastic waste management implemented nowadays namely landfill, incineration and energy recovery, downgauging, re-use of plastic packaging and plastic recycling. The waste plastic could be diverted from lettering and entering the landfill by packaging downgauging or substitution of heavy packaging with lighter ones and designing products that is able for reusing, repairing and re-manufacturing. The waste volume could be reduce by reducing the amount of packaging used per item. Later, the recycling is the answer once the waste enters the landfill. Moreover, the organic waste namely plastic could be used for energy recovery but less desirable due to the release of hazardous substances during the process. Energy-recovery processes may be the most suitable way for dealing with highly mixed plastic. In order to decrease the environment desirability, the 4Rs strategy is obtained which is reduce, reuse, recycle (waste) and recovery (energy) with landfill is the least desirable in management strategy. However, the least desirable in managing waste is the most preferable by the community nowadays.
6. Another vital concern is an increasing amount of plastic waste materials at the landfill that are generated from the plastic industry. Gürü et al. (2014) stated that plastics are used by almost all end-use segment of the economy and the increase of the development in the plastic industry may lead to the increase of plastic wastes. Moreover, compared to other wastes, plastics wastes could not be decomposed and thus occupies the volume of the landfill for a long period

which may cause plastic wastes to mount up in the landfill. The waste plastics are possible to be recycled instead of being disposed at landfill sites in which the recycling way is more financially attractive.

The use of recycled plastics as a bitumen modifier could enhance the commercial viability of the recycling process and at the same time promote sustainable approach towards waste management (Nassar et al. 2012). Therefore, the needs still exist to solve these problems for developing more effectives PMB that is greatly enhancing the properties of the binder with a reasonable cost.

1.3 Aim and Objectives of Research

The main aim of this research is to investigate the advantages of using recycled waste plastic (RWP) as a modifier without adding any compatibilising groups in bitumen in terms of its performance and also to develop a better understanding on PMB. The types of RWP that will be used in this study are Polyethylene Terephthalate (PET), High Density Polyethylene (HDPE), Polyvinyl Chloride (PVC), Low Density Polyethylene (LDPE), Polypropylene (PP) and Polystyrene (PS).

In order to achieve the aim of the research, the main objectives of this research can be summarised as follows:

1. To identify and optimise the potential of RWP to be used as a bitumen modifier.
2. To understand and investigate the different factors that affects the RWP-modified bitumens.

3. To investigate the effects of modifying bitumen with RWP on the rheological properties and the performance characteristics of binder and asphalt mixture at intermediate and high temperatures.
4. To evaluate the durability of RWP-modified bitumen that exposed to moisture and ageing conditions.

1.4 Research Methodology

In this research, the research methodology consists of three main phases and Figure ***1.1*** illustrated the test involved for each phases.

1.4.1 Phase 1: Selection and Incorporation of RWP in Bitumen

The main objective of the first phase is to identify the potential of RWP as a modifier in bitumen and to produce the most promising and appropriate blending condition of polymer modified bitumen that is qualified to be used for second phase. In the first phase, the work focuses on selecting RWP by means of polymer types and optimising the blending parameters Polymers that cannot be blended with bitumen will be excluded from the testing program and the most promising blending parameters will be used to produce PMB for further testing in Phase 2.

1.4.2 Phase 2: Binder Test

After optimising the blending condition of RWP-modified bitumen, the binder performance tests will be performed in Phase 2. The objective of this phase is to identify which RWP will improve the performance of modified bitumen at different level of in-service temperature. In order to develop better understanding on the modification of bitumen, the modification processes associated with different RWP-modified bitumen blended will be comprehensively evaluated and several tests will be carried out involving chemical, structural, rheological, and performance analysis.

Then, all of these results will be considerate together and the most promising RWP-modified bitumens with optimum RWP content will be obtained and used in Phase 3.

1.4.3 Phase 3: Mixture Performance Test

The main objective of this phase is to investigate the effect of adding RWP-modified bitumen in asphalt mixture based on performance characteristics of asphalt mixture. This will be achieved through a series of mechanical performance testing of asphalt mixture.

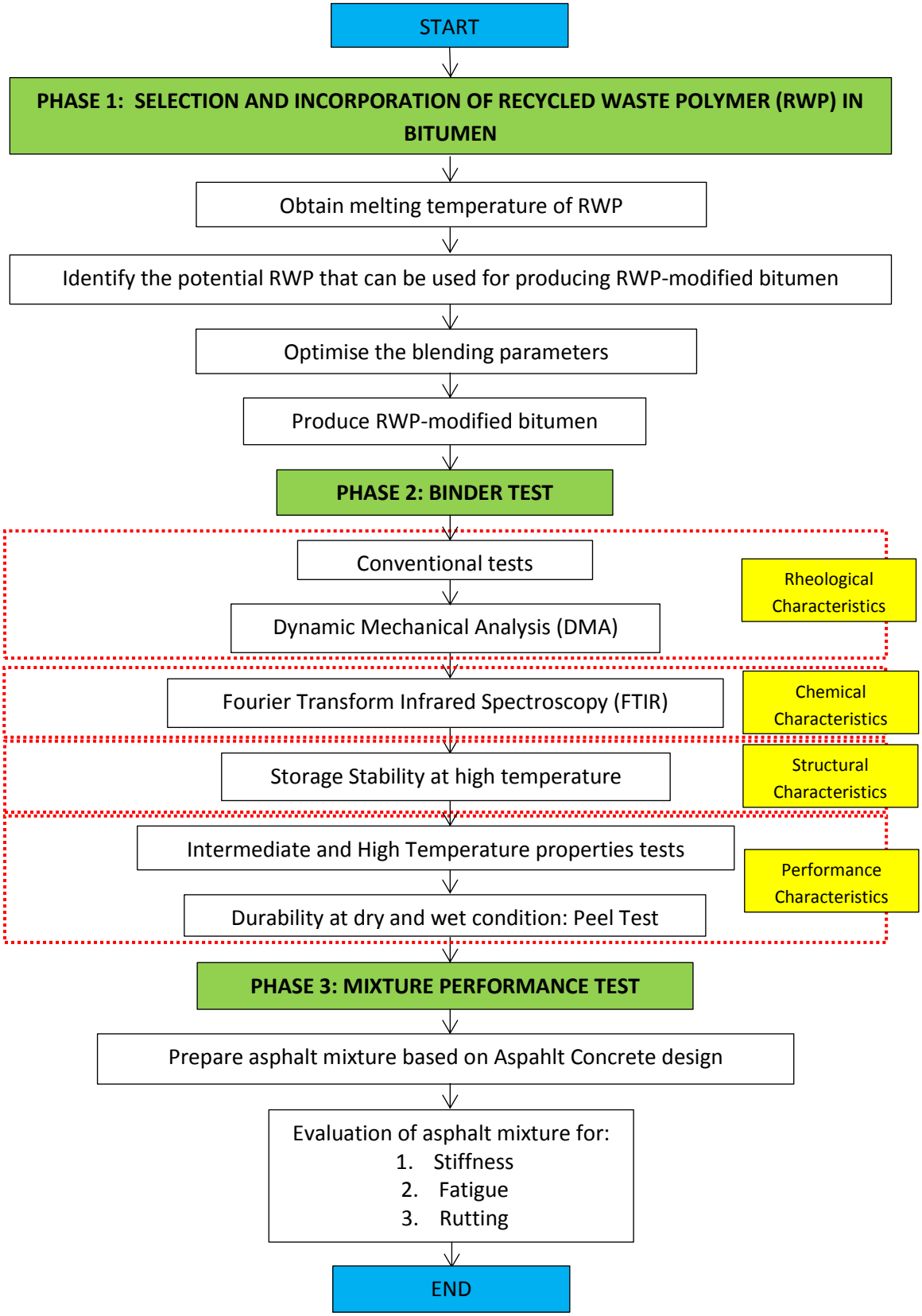


Figure 1.1: Workflow chart

1.5 Thesis Structure and Outline

Chapter 1 represents the general perspective related to this study including the research background, problem statements, the aim and objective that need to be achieved and the research methodology.

Chapter 2 gives a review on history of polymer modified bitumen. The basic concept of modified bitumen and road pavement are explained. The previous studies of using polymer to enhance the binder properties also included.

Chapter 3 deals with the details of the laboratory experimental work involved in this study. It begins with a description of the materials used and then followed by the experimental setups for binder tests, manufacturing RWP-modified mixture and asphalt mixture tests.

Chapter 4 describes in detail the procedure for selecting RWPs that are suitable to modify base binder. The most promising and appropriate blending parameter of RWP-modified binder is obtained and used to produce modified binder. Lastly, the chemical and structural characterisations of the modified binders were examined.

Chapter 5 presents the results of rheological and performance properties of the selected RWP-modified binders. Besides that, the effect of RWP addition on ageing was investigated in terms of its rheological properties. Then, the most promising percentage of RWP in enhancing these properties were selected and used for further analysis.

Chapter 6 covers the details of further comparison between the most promising percentage of RWP with another two additional binders (act as a conventional

binder) in terms of mechanical properties, rheological properties, fatigue cracking, fracture resistance and rutting.

Chapter 7 investigates the effect of adding RWP on durability of the binder at dry and wet conditions by means of the Peel test. The fracture energy and types of failure were used to evaluate the effect of moisture on aggregate-bitumen bonds.

Chapter 8 provides a study of asphalt mixture performance of modified binder. The performance of asphalt mixtures was characterised in term of the stiffness modulus, resistance to fatigue and resistance to rutting by means of ITSM, ITFT and RLAT, respectively. Two binder contents namely 5% and 6% were used for comparison.

Chapter 9 outlines the conclusions obtained from this study and the recommendations for further study.

CHAPTER 2: **LITERATURE REVIEW**

2.1 Background

Bitumen has been known to be used as a binder for road construction and plays a vital role for binding aggregate together which is by coating over the aggregate. However the performance of this bituminous binder is questioned time and again, given that they are brittle and hard in cold environments and soft in hot environments (Behl et al. 2014). From a rheological point of view, bitumen should be considered a viscoelastic material, very sensitive to temperature (Yu et al. 2015), because it shows a predominant elastic behaviour at low temperature and viscous characteristics at moderate and high temperatures (Pérez-Lepe et al. 2003). Due to the growing levels of traffic load on roads and changeable weather conditions, research to improve the properties bitumen has become increasingly important (Fang et al. 2014b). The pavement performance can be improved by adding modifier into the original bitumen to change the components, structure and the viscoelasticity of bitumen (Fang et al. 2014a). Brule (1996) stated in his study that an ideal modified binder should have enhanced cohesive and very low temperature susceptibility throughout the range of temperatures to which it will be subjected in service, but low viscosity at the usual temperatures at which it is placed. He added that the susceptibility to loading time should be low, whereas its permanent deformation resistance, breaking strength and fatigue characteristics should be high and at the same time, it should have at least the same adhesion qualities as traditional binder. Brown et al. (2009) stated that the modifications of bitumen should be modified to achieve the following improvements:

1. Lower stiffness (or viscosity) at high temperatures during construction to facilitate pumping the binder as well as mixing and compaction asphalt mixture.
2. High stiffness at high temperatures (summer season) for reducing rutting and shoving.
3. Lower stiffness and faster relaxation properties at low service temperature for increasing the resistance of thermal cracking.
4. Increased adhesion between bitumen and aggregate in the presence of moisture to reduce stripping.

Virgin polymers have been used for a number of years for the modification of asphalt concrete mixtures because most of them have significant performance and economic implications and certain polymers may offer some help in improving the asphalt pavement problems such as stripping, permanent deformation, flushing, thermal cracking, etc. (Al-Hadidy & Tan 2009a). However, virgin polymers are uneconomical when used as a modifier because of their high cost and thus, only small quantity can be used to improve road pavement (Kalantar et al. 2012).

Currently, the use of waste polymer is being considered for bitumen modification due to the high cost of the virgin polymer (Köfteci et al. 2014) and massive increase in production of various types of waste plastics. In fact, the world production of plastic in 2018 is about 390 million metric tonnes with the increase of 9% per year since 1950 (Ryberg et al. 2018). From the total global plastic production, the new plastic material is produced around 360 million tonnes (Garside 2019) and the remaining 30 million tonnes is from recycled plastics. Lebreton and Andrady (2019) stated that in the next 20 years, the plastic production is estimated to be

double according to the current rate of growth. At the end of their service life, the plastics become waste that later will be collected and treated. Unfortunately, only 173 million tonnes of plastic wastes are collected whereby 50 million tonnes is used for recycling, 72 million tonnes end up in the landfills and 51 million tonnes is incinerated for energy recovery. While the huge amount of plastic wastes is disposed improperly through illegal dumping and leaks into the environment.

Polymer products are widely used in daily life so that the cost of this polymer is modest and as a recycled material the cost is even lower (Yu et al. 2015). This is the reason why many researchers have focused on the use of cheaper materials for bitumen modification (Polacco et al. 2005). Therefore, the use of waste plastics for replacing virgin polymers as a modifier is a worthy concept, which may result in greater cost savings (Fang et al. 2014b). Gawande et al. (2012) stated that the common method of modifying the rheological properties of bitumen is blending with synthetic polymers like rubber and plastics and the use of plastic waste is parallel to polymer modified bitumen. Theoretically, polymer modified bitumen (PMB) can be produced when incorporating between polymer and bitumen by using mechanical mixing or chemical reaction (Lu et al. 2011).

2.2 Asphalt Pavement

Pavement are layered structures whereby each layer of pavement is designed as a medium to transfer the applied vehicle loads to foundation soil. Generally, there are two type of pavement namely flexible pavement and rigid pavement. These pavements are categorized based on their structural type and differentiated through on how the load is distributed to the subgrade. In flexible pavement, the wheel load stresses are transferred to the lower layers by grain-to-grain contact of the aggregate

through the granular structure while rigid pavement will transmit the wheel load stresses to subgrade soil by flexural strength of the pavement. In this study, the modified binder was used for flexible pavement and thus, only this type of pavement is discussed in this section.

The wheel load applied on the flexible pavement will be distributed to a wider area and the stress decreases with the depth. Basically, flexible pavements are constructed of several layers in which the top layer is the strongest material (least flexible) for sustaining maximum compressive stress while the weakest material (most flexible) is used at lower layer because this layer experienced lesser magnitude of stress. The structure of flexible pavement consists of surface course, binder course, base course, subbase course and subgrade as shown in Figure 2.1. Besides that, three types of coats are used in the flexible pavement system which are seal coat, tack coat and prime coat.



Figure 2.1: Typical cross section of flexible pavement

- **Surface course:** Surface course is also known as wearing course is a bituminous layer that contains high quality material to withstand direct loading from the traffic. The main function of this layer is to provide skid resistance surface which

vehicles can travel safely, friction and drainage for the pavement. The surface course is constructed using a good quality aggregates and high dense bitumen.

- **Binder course: T**he binder course consists of aggregates and bitumen. The material used in this layer is similar to the base course but smaller aggregate size with less bitumen content than surface layer. The purpose of binder course is to transfer the loads from the surface course to the base course.
- **Base course:** Base course is constructed either hydraulically bound (e.g. concrete) or granular or both with larger aggregate size. Apart from structural support, the function of this layer is to distribute the loads from the top layers to the subbase and subgrade layer. Moreover, the sub surface drainage system can be provided within the base course.
- **Subbase course:** Subbase course is placed beneath of base course and the function of this layer is similar between these two layers. Generally, the hydraulically bound or granular is used in this layer and the construction of subbase course is dependent on the subgrade soil. A subbase course is not needed if the soil of subgrade is strong and stiff.
- **Subgrade:** Subgrade layer is a foundation for the pavement system that located at the bottom layer. This layer consists of a natural soil layer that is compacted to the desired density. The main purpose of subgrade is to transfer the load from the above layers to the ground and thus, it should be strong enough to take the stresses.
- **Seal coat:** Seal coat is a thin surface treatment used to waterproof the surface course and to provide skid resistance.

- **Tack coat:** Tack coat is a very light application of asphalt, usually asphalt emulsion diluted with water. It provides proper bonding between two layers of binder course and must be thin, uniformly cover the entire surface and set very fast.
- **Prime coat:** Prime coat is an application of low viscous cutback bitumen to an absorbent surface like granular bases on which binder layer is placed. It provides bonding between two layers. Unlike tack cost, prime coat penetrates into layer below, plugs the voids, and forms a watertight surface.

2.2.1 Pavement Deterioration

The major flexible pavement failure modes are fatigue cracking, rutting and thermal cracking. On the road pavement, the passing wheel load could induce the bending of pavement layers that later will cause horizontal tensile stresses and strains at the bottom of each bound layers and vertical compressive stresses and strain in the bulk section as shown in Figure 2.2. The repetitive wheel loading can cause fatigue cracking at the bottom of the bound layer and rutting of the vertical section of the layer. When exposed to temperature changes particularly at low temperatures, the asphalt mixtures undergo thermal expansion and contraction. Due to the layered structure of the pavement, the thermal contraction is restrained by the friction with other layers in contact particularly in the longitudinal direction could build up the stress and cause thermal cracking. Besides that, the repeated strain caused by thermal contraction and expansion could result in thermal fatigue.

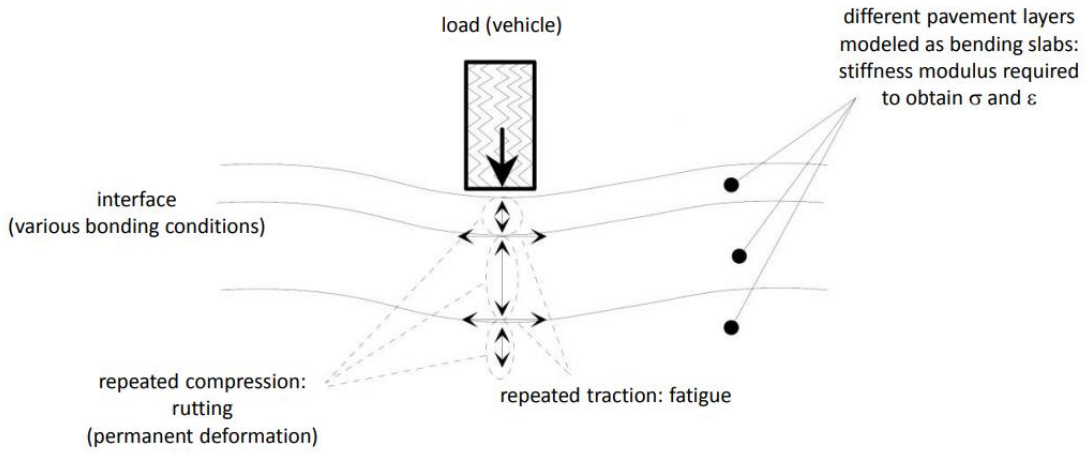


Figure 2.2: Traffic load and pavement response

2.2.1.1 Fatigue cracking

Fatigue cracking is one of the main distresses of a pavement structure that occurs at intermediate temperatures between 10 to 40°C. This distress is caused by repeated loading whereby the crack will appear and degrade the bound pavement materials and then propagate into the pavement structure as shown in Figure 2.3 (Cocurullo et al. 2008). Pavement is considered failed if 20% of the surface has cracked.



Figure 2.3: Example of fatigue cracking

2.2.1.2 Rutting

At high pavement temperature conditions which are more than 40^{o}C whereby the binder tends to become softer and more viscous, the major failure mode exhibited by the flexible pavement is rutting or permanent deformation (Figure 2.4). Rutting appears as a result of the increasing number of traffic load repetitions which cause the plastic deformation of the materials of each layer in the pavement structure. Rutting can also be caused by consolidation of the pavement layers by traffic after construction has been completed. If the asphalt pavement is not properly manufactured and compacted, rutting can occur from the lateral plastic flow of the pavement layer caused by the high force of radial truck tires. The used of higher binder content could lead to this phenomenon due to the loss of internal fraction between aggregate particles. Rutting can also be the result of using rounded aggregate. The road pavement is considered failed if it exhibits a rut depth of 20 mm.



Figure 2.4: Example of rutting

2.2.1.3 Thermal cracking

Thermal cracking is the crack that is relatively perpendicular to the centreline of the pavement (Figure 2.5) and primarily a result of the low temperature environment. This thermal crack phenomenon is related to the relaxation capability of binders. It starts at the surface and progresses down with time. This happens due to the low ambient temperatures chill the road surface first. The road pavement subjected to high cooling rates and low temperatures develop tensile stresses due to shrinkage. If these stresses exceed the fracture strength of the pavement layer, transverse cracking develops. Asphalt mixtures which has high stiffness at low temperatures are susceptible to cracking.



Figure 2.5: Example of thermal cracking

2.3 Constituent Materials

2.3.1 Bitumen

In BS 3690, Part 2: 1989, bitumen is defined as 'A viscous liquid, or a solid, consisting essentially of hydrocarbons and their derivatives, which is soluble in trichloroethylene and is substantially non-volatile and softens gradually when heated. It is black or brown in colour and possesses waterproofing and adhesive properties. It is obtained by refinery processes from petroleum and is also found as a

natural deposit or as a component of naturally occurring asphalt, in which it is associated with mineral matter'.

Basically, the constituent (chemical) and structural (physical) composition of bitumen composed of hydrocarbons namely chemical combination of carbon and hydrogen but the amount of hydrocarbons varies and the chemical compositions also vary which is depending on the crude source (Brown et al. 2009). Besides hydrocarbon molecules as its large constituent, bitumen also composed of small functional groups of non-hydrocarbons that is known as heterocyclic atoms (McNally 2011; Nassar et al. 2011) which are sulphur, oxygen and nitrogen (Romberg et al. 1959; Read & Whiteoak 2003). The average quantity of these molecules are as follows:

- Carbon: 82-88%
- Hydrogen: 8-11%
- Oxygen: 0-1.5%
- Sulphur: 0-6%
- Nitrogen: 0-1%

Bitumen can be classified into two groups called asphaltenes and maltenes which are constituted according to their solubility in hexane and heptane (Ait-Kadi et al. 1996). Asphaltenes have high molecule weight components that are insoluble in hexane or heptane and high in polarity while maltenes are the low molecular weight and soluble in those solvents (Glover 2007). Maltenes are composed of saturates, resin and aromatics compound (Corbett 1970; Lesueur 2009). The proportion of asphaltenes to maltenes is vital in determining whether the bitumen is sol-type or

gel-type bitumen as shown in Figure 2.6. Sol-type bitumen generally will behave like Newtonian fluids while gel-type bitumen show completely elastic after being slightly deforms (Bonemazzi & Giavarini 1999).The main constituent of bitumen are described as follow:

- **Asphaltenes:** are dark brown to black friable solids that contain relatively large amounts of heteroatoms, such as O, N and S, as well as metal ions like nickel, vanadium and iron. The structure of asphaltenes may contain an aromatic ring system with short side chains. Asphaltenes are the highest molecular weight ranging from 1,000 g/mol to 100,000 g/mol with a particle size of 5 mm to 30 mm which contribute around 5 to 20% of the bitumen mass. The amount of asphaltenes greatly affects the rheological properties of bitumen whereby the bitumen with high asphaltenes content will produce bitumen that is hard, higher softening point and more viscous.
- **Resins:** Resins are dark-coloured, heavy, semisolid or solid and very adhesive. They are fluid when heated and become brittle when cold. Resins are the dispersing agents or peptiser for asphaltene colloidal system to provide a homogeneous liquid. The molecular weight of resins is in the range of 500 g/mol to 50,000 g/mol and form about 13% to 25% of the bitumen mass.
- **Aromatics:** Aromatics are black or brown in colour and contain the lowest molecular weight compound in the bitumen that is range between 300 g/mol and 2,000 g/mol and is the major proportion in the bitumen mass ranging about 40% to 65%. In the bitumen, aromatics acts as a plasticiser and also a solvent for

asphaltenes and resins. During ageing process, aromatic could help in protecting the oils from oxidation (Read & Whiteoak 2003).

- **Saturates:** Saturates are white in colour and non-polar viscous oil (give fluid properties to the system) that constitute 5% to 20% of overall bitumen mass with the molecular weight range between 300 g/mol and 1,500 g/mol (quite similar with aromatic molecular weight). Softer bitumen which might able to reform broken bonds generally has more saturates.

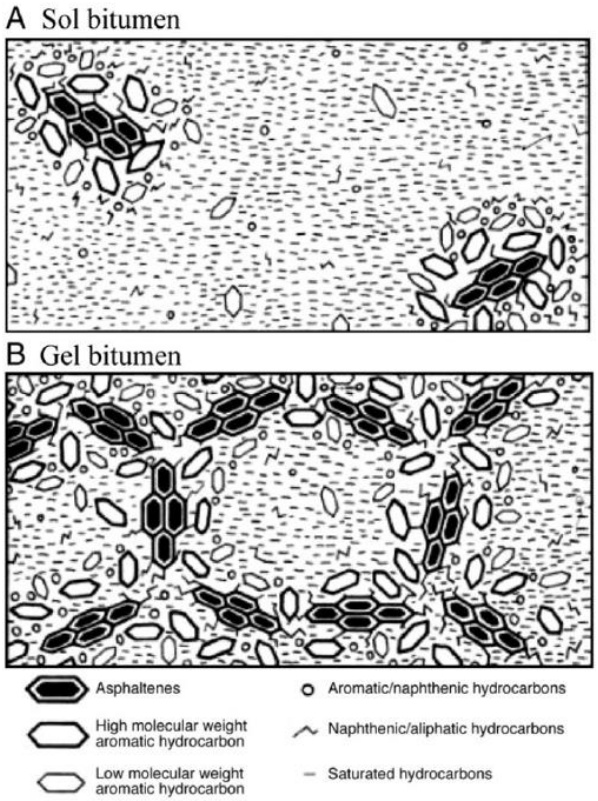


Figure 2.6: Schematic representation of a) SOL type, b) GEL type bitumen structure (Read & Whiteoak 2003)

Generally, in the soft bitumens could resist fatigue cracking due to its flexibility but susceptible to permanent deformation meanwhile, the hard bitumen could perform better in rutting resistance due to its high stiffness but may be sensitive to the fatigue damage (Jiménez del Barco Carrión 2017; Moreno-Navarro et al. 2017).

2.3.2 Recycled Plastic

In plastics world, there are twenty or more known basic types of plastics but generally, it's specified into several groups namely:

1. Cellulose Plastics.
2. Synthetic Resin Plastics.
3. Protein Plastics.
4. Natural Resins.
5. Elastomers
6. Fibers

Those above mentioned groups could be classified as thermoplastic and thermosetting materials whereby it's depending upon their physical properties when exposed to the heat. The materials that can be shaped into desired shapes in new models under heat and pressure and then become solids when cooling is called as thermoplastic. The thermoplastic materials can be remoulded if the same conditions of heat and pressure are applied. The other type is called thermosetting materials which is this materials cannot be softened and redesigned when applying heat once it is shaped (it does not withstand heat) (Gawande et al. 2012; Sulyman et al. 2014). Recent study has been conducted by Köfteci et al. (2014) noticed that the physical properties of recycled plastics used for modification will influence the hardening of modified bitumen. Table 2.1 shows the examples of some thermoplastic and thermosetting materials.

Table 2.1: Examples of Thermoplastic and Thermosetting Materials (Gawande et al. 2012; Sulyman et al. 2014)

Thermoplastic	Thermosetting
Polyethylene Teryphthalate (PET)	Bakalite
Polypropylene (PP)	Epoxy
Polyvinyl Acetate (PVA)	Melamine
Polyvinyl Chloride (PVC)	Polyester
Polystyrene (PS)	Polyuryurethane
Low Density Polyethylene (LDPE)	Urea-Formaldehyde
High Density Polyethylene (HDPE)	Alkyd

In the pavement field, there are four group of waste materials are used namely industrial wastes, domestic wastes, mineral waste and agricultural wastes. For the recycled plastics waste, it falls under domestic group and can be classified into 6 major types as followed:

1. Polyethylene Teryphthalate (PET)

PET is a thermoplastic polymer resin that come from polyester family and generally used in synthetic fibers, beverage, food and other liquid containers, thermoforming applications and engineering resins with combination of glass fiber. It produced from the process of polymerization of colourless liquid ethylene glycol (obtained from ethylene) and crystalline solid of terephtalic acid (obtained from xylene) that have been heated with the presence of chemical catalyst. Then, the end-product of the process produce PET in the form of molten and viscous mass whereby it will be solidified to form a plastic (Modarres & Hamedi 2014).

2. Polypropylene (PP)

Polypropylene is the lightest polymer in the industrial polymer that produced by polymerization of propylene. It has good heat and chemical resistance, high stiffness, surface hardness and toughness at normal temperature and resistance to deformation at elevated temperature (Sadeque & Patil 2013).

3. Polyvinyl Chloride (PVC)

Polyvinyl Chloride can be derived from salt and oil. Chlorine is manufactured by the electrolysis of sodium chloride (salt). The chlorine is then combined with ethylene that has been obtained from oil. The resulting element is ethylene dichloride, which is converted at very high temperatures to vinyl chloride monomer. These monomer molecules are polymerized forming polyvinyl chloride resin. The characteristics of PVC are toughness, strength, ease of blending and processing, flame resistance and have fire prevention properties.

4. Polystyrene (PS)

Polystyrene is a plastic which has been extensively used in packaging and offer shockproof material in many fields (Fang et al. 2014a). It is a petroleum based plastic made of the styrene monomer. Polystyrene are durable, lightweight and thermal insulating.

5. Low Density Polyethylene (LDPE)

LDPE resins are thermoplastics made from the polymerization of ethylene at very high pressures. For a short time, it can withstand temperatures of 80°C continuously and 95°C. LDPE is a semi-rigid material that are very tough, weather proof, good chemical resistance, low water absorption, easily processed by most methods and low cost.

6. High Density Polyethylene (HDPE)

High Density Polyethylene is produced from ethylene by a catalytic process. The absence of branching results in a more compact structure with a higher density and somewhat higher chemical resistance than LDPE. HDPE can be classified as flexible, waxy, weatherproof, good low temperature toughness (up to -60^{o}), easy to process by most methods, low cost, good chemical resistance.

In 1988, the Society of the Plastics Industry (SPI) was developed the SPI resin identification coding system as illustrated in Table 2.2. The code is a set of symbols placed on plastics to classify the polymer type that will be used during separation of different polymer types for recycling purpose.

Table 2.2: Resin Codes and Examples of Each Recycled Materials

Waste Plastic	Resin Identification Code Image	Properties	Applications
Polyethylene Teryphthalate (PET)		• Clear, tough, solvent resistant, barrier to gas and moisture • Softening range: 90^{o}C (approx.) • Melting range: 250^{o}C to 260^{o}C (approx. for crystalline)	Drinking water bottles, tote bags, furniture, carpet
High Density Polyethylene (HDPE)		• Hard to semi-flexible, resistant to chemicals and moisture, waxy surface, opaque,	Carry bags, bottle caps, household articles

		easily coloured, processed and formed • Softening range: 80° to 90°C (approx.) • Melting range: 110° to 140°C (approx.)	
Polyvinyl Chloride (PVC)		• Strong, tough, can be clear, can be solvent welded. • Softening range: 80° to 90°C (approx.) • Melting range: 240 (approx.)	Mineral water bottles, credit cards, toys, pipes and gutters; electrical fittings, furniture, folders and pens, medical disposables
Low Density Polyethylene (LDPE)		• Soft flexible, waxy surface, translucent, scratches easily • Softening range: 80° to 90°C (approx.) • Melting range: 120°C (approx.)	Carry bags, sacks, milk pouches, bin lining, cosmetic and detergent bottles
Polypropylene (PP)		• Hard and translucent, translucent, withstands solvents, versatile. • Softening range: 95° to 110°C (approx.) • Melting range: 140° to 160°C (approx.)	Bottle caps and closures, wrappers of detergent, biscuit, vapors packets, microwave trays for readymade meal

Polystyrene (PS)		• Clear, glassy rigid, opaque, semitough, affected by fat, acids and solvents, but resistant to alkalis, salt solutions, Low water absorption, when not pigmented is clear, is odour and taste free. • Softening range: 80^{o} to 90^{o}C (approx.) • Melting range: 240 (approx.)	Yoghurt pots, clear egg packs, bottle caps. Foamed Polystyrene: food trays, egg boxes, disposable cups, protective packaging

2.4 History of Polymer Modified Bitumen (PMB) (Becker et al. 2001)

Virgin and synthetic polymer have been used as a modifier in bitumen as early as 1843. In the 1930s the project was underway in Europe and North America began to use rubber latex in 1950s. Europe was using modified asphalts ahead of the United States which were limited to use PMB because of its high expenses in the late 1970s. In the mid-1980s, US began to use new developed polymers and European technologies. Currently in Australia, polymer modified binders is included in the guides and specifications of National Asphalt Specification. In the survey of state departments of transportation in 1997, 47 states of US reported that in the near future they would be using modified asphalts and 35 states reported that they would need bigger portions. Several investigations all around the world have researched and evaluated benefits of modifying polymers on the performance of pavement and developing the specifications and tests for binders are still continuing.

Over the last decade, USA is the country where most of the research is done, followed by China, Canada and some European countries. Among the companies that have been filing patents on PMB over the last decade, Marathon Ashland Petroleum LLC is the leading one. The Goodyear Tire and Rubber Company, Fina Technology, Polyphalt LLC, BASF Corporation and Ergon Incorporated are also reported. There have been significant movements in the marketing area. The interest for PMB technology has been increasing, and so the number of companies which commercialize it, and their earnings. The United States, China, France and Italy are leaders in PMB research and development activities, even though considerable work has also been done in Japan, Germany, Russia, Great Britain, and Canada.

2.5 The Benefits of Using Polymer in Bitumen

In the last decade, polymer modified bitumen has been used to improve the quality of bitumen. A significant number of research works have been carried out on PMB (Lu & Isacsson 1997a; Al-Dubabe et al. 1998; Isacsson & Lu 1999; Lu & Isacsson 2000; DomíNguez 2011; Gürü et al. 2014). It has been recognised that the deficiencies of bitumen can be overcome by the addition of polymers for improving viscoelastic behaviour besides maintaining its own advantages (Behl et al. 2014). Polymers can significantly improve the asphalt pavements performance at low, intermediate and high temperatures and modifying bitumen with even small amounts of polymers may increase the road pavement life span (Kalantar et al. 2012). They can increase the resistance of mixture to permanent deformation, thermal fracture and fatigue cracking at low temperature, decrease plastic flow and increase shear modulus at high temperature (González et al. 2002; Pérez-Lepe et al. 2003). Besides that, substitution of bitumen with polymer in paving and maintenance application is

possible to be done due to its improvement in engineering properties including thermal cracking, stripping, rutting resistance, temperature susceptibility and fatigue damage (Kalantar et al. 2012).

Since PMB has been widely used for paving application due to its promising benefits. As the production of polymer modified asphalt pavement is increased, the impact of using polymer on the recyclability of the pavement is essential to be investigated. However, only a few studies have been conducted regarding the recycling of polymer modified asphalt pavement. (108_printed) investigated the potential of PMB for recycling on the eight years old PMB extracted from a wearing course mixture of road sample taken from US61 highway in Livingston Parish, Louisiana and reported that no residual polymer is detected in the modified binder due to the extensive field ageing which may cause the polymer to be degraded and disappear. The recovery PMB showed higher age hardening and brittle while the performance test of asphalt mixture containing recycled PMB exhibited increase the rutting resistance but decrease the fatigue resistance as the amount of recycled PMB increase in the mixture. Later, (109_printed) suggested that the additives should be added to the recycled PMB for achieving the desired rheological properties.

2.6 Incorporation of Polymer into the Bitumen

Use of polymer in bitumen for asphalt mixtures generally has two common approaches namely wet process and dry process. Table 2.3 summaries the advantages and disadvantages of wet and dry process.

In the wet process, the polymer is blended with bitumen at the particular temperature with the proper mixer until achieving a homogeneous blend before

being used in the pavement mix. The two main parameters namely blending temperature and blending time will depend on the type of polymer and bitumen used (Kalantar et al. 2012). Shell stated that the mixing temperature should not exceed 185^{o}C in order to avoid the bitumen from burning and the mixing time should be optimized to produce homogenous dispersion of the polymer in the bitumen (Lu & Isacsson 1997a; Kalantar et al. 2012). Besides that, Gawande et al. (2012) reported the use of polymer in bitumen by using wet process will increase the melting point of the bitumen and during winters, the polymer modified bitumen makes the road retain its flexibility that would leads to its long life. This happened because of the polymer acts as a strong 'binding agent' for the bitumen. It also could withstand adverse soaking conditions under water for long period.

Dry process consists of blending the polymer with the aggregate in the solid form prior the addition of the bitumen. Coating the aggregate with the polymer after which bitumen will then be added could improve the aggregate quality in term of voids, moisture absorption and soundness.

Table 2.3: Advantages and Disadvantages of Wet and Dry Process (Gaweł et al. 2011; Gawande et al. 2012)

Type of Process	Advantages	Disadvantages
Wet Process	• Can be utilized for recycling of any type, size, shape of waste material (Plastics and Rubber)	• Time consuming- more energy for blending. • Powerful mechanical is required. • Additional cooling is required as improper addition of bitumen may cause air pockets in roads.

		• Maximum amount of waste plastic can be added around 8 %.
Dry Process	• Plastic is coated over stones - improving surface property of aggregates. • Coating is easy & temperature required is same as road laying temperature. • Use of waste plastic more than 15% is possible. • Flexible films of all types of plastics can be used. • Doubles the binding property of aggregates. • No new equipment is required. • Bitumen bonding is strong than normal. • The coated aggregates show increased strength. • As replacing bitumen to 15% higher cost efficiency is possible. • No degradation of roads even after 5-6 years after construction. • Can be practiced in all type of climatic conditions. • No evolution of any toxic gases as maximum temperature is 180^{o}C.	• Applicable to plastic waste material only • Limited application compared to wet process • Swelling problem

2.7 Factors that Influence the Properties of PMB

2.7.1 Bitumen Characteristics

The nature of bitumen is prominently influenced by the binder micromorphology, mechanical properties and the stability of the blend. In order to improve the binder performance at different in-service temperature, the polymer must be compatible with the bitumen and maintain its compatibility during storage and usage. Due to the different in molecular weight and structure, viscosity and density of the PMB constituents, compatibility between polymer and bitumen become difficult to accomplish (Giavarini et al. 1996). Besides that, the production process of the original crude also affects the PMB compatibility. The obtained results may vary if bitumen from other sources is used (Al-Hadidy & Yi-Qiu 2009b). However, the well PMB blends can be obtained when the bitumen contain enough oil fractions to dissolve and expand the polymer (Kalantar et al. 2012).

When the polymer content is low, the continuous bitumen phase is enriched with resins and asphaltenes, resulted increase in the consistency and the elastic properties of the binder (Becker et al. 2001). Commonly, thermoplastic polymer modified asphalt which resulted from physical mixing of the constituents without chemical interactions, can produce a two-phase system whereby one phase is a swollen polymer and another phase grouping the constituents of the asphalt not intervening in the solvation. The physical properties of PMB blends will significantly change if the polymer content is increased. Thus, significant increase in the plasticity interval, in tensile strength and elastic properties, and reduction in thermal sensitivity can be observed (Kalantar et al. 2012).

Low content of asphaltenes in bitumen may produce a single phase blend of PMB because the ability of the polymer to absorb more maltenes is high if the percentage of asphaltenes is low (Becker et al. 2001). The permissible level of asphaltenes concentration is depending upon: 1) polymer content, 2) polymer molecular weight, 3) asphaltenes molecular weight, and 4) aromaticity. It is vital to balance the aromatic content for producing stable PMB and make the polymer as continuous phase for achieving the maximum possible benefits of the polymer. If the bitumen is high in aromaticity, the polymer can be solubilized (Brûlé et al. 1988).

2.7.2 Polymer Characteristics

In PMB, an effective polymer must increase the resistance of rutting at high temperature without making it too viscous for blending procedure or too brittle at low temperature and able to be processed with bitumen by using conventional equipment. The polymer should be sufficiently compatible with the bitumen for avoiding phase separation during the storage, transportation, application and service and also cost effective for reducing road costs. There are several factors that affect the PMB process which are polymer content, chemical composition, structure, average molecular weight, molecular weight distribution, degree of branching, crystallinity, etc. (Giavarini et al. 1996; Becker et al. 2001).

2.8 Compatibility of PMB

Compatibility is defined by the extent of segregation of the bitumen and polymer during hot storage (140-180^{o}C) (Pérez-Lepe et al. 2006). PMB blend tends to show a two-phase morphology, depending on the polymer type, concentration, base bitumen composition and the aromaticity (lower molecular weight polar naphthenic), the system can have an bitumen-rich phase with a homogeneous dispersion of

polymer spheres or continuous polymer-rich phase with dispersion of asphaltene globules in it (Sybilski 1997; Habib et al. 2011). A proper degree of compatibility between bitumen and polymer is vital in order to avoid phase separation occur during storing, pumping and application of the binder for achieving desired properties of the pavement.

With regard to the latter, the low compatibility between bitumen and polymer can lead to phase separation when the material is stored at a high temperature (160 to 200^{o}C) in the absence of stirring (Al-Hadidy & Yi-Qiu 2009c) because the polymer-rich phase migrates to the higher part of the storage tank, while an bitumen-rich phase segregates into the lower part. Reduced in storage stability will produce unsuitable PMB for paving because of unfailing modifies its structure and consequently its properties. However, by storing the modified bitumen samples at low temperature after preparation may prevent segregation and sedimentation because the molecular chains of the polymer and bitumen were frozen (Yu et al. 2015).

There are some factors that affect the compatibility of PMB which are (Lesueur 2009): 1) High asphaltenes content in bitumen will decrease the polymer/bitumen compatibility, and 2) The aromaticity of the maltenes needs to fall between certain values to achieve a good level of compatibility. It is demonstrated that the stability of polymer modified bitumen depends not only on the difference in density and viscosity between bitumen and polymer (Pérez-Lepe et al. 2007) but also on bitumen structure and bitumen/polymer molecular interactions (Pérez-Lepe et al. 2006). Isacsson and Lu (1999) found out that better dispersion of the polymer was observed for the modified binders when using a base bitumen with higher content of aromatic

component and low content of asphaltenes and thus resulted, better compatibility and higher storage stability of PMB. While mixing either using mechanical or chemical method, the differences in molecular weight and polarity of base bitumen and polymer has critical effect on compatibility (Lu & Isacsson 1997b; Habib et al. 2011). In order to stabilize the polymer particles within the bituminous matrix, an effective mixing process should be achieved (Pérez-Lepe et al. 2007). There are three types of compatibility that will occur during modifying the bitumen which are (Brule 1996; Fang et al. 2008):

1. **The PMB is heterogeneous**

- This kind of PMB can be considerate as incompatible because when mixing the polymer with bitumen, it results in heterogeneous mixture without cohesion or ductility. The constituents in the binder will separate and produce PMB that has none of the characteristics of a road binder. It was reported that the addition of polymer under agitation at high temperature generally results in asphaltene flocculation and oil bleeding, resulting a binder with no cohesion.

2. **The PMB is totally homogeneous**

- This is the uncommon case of compatibility whereby it happened when the oil in the bitumen solvate the polymer perfectly and terminate any intermolecular interactions. The quality of modification with respect to those of the initial bitumen is very slight although the binder exhibits stable blends because it only increases the viscosity which is undesirable.

3. **The PMB is micro-heterogeneous**

- This type of compatibility is desirable because yield a stable blend physically. The mix is made up from two distinct finely interlocked phases, resulting better

properties of modified bitumen. In the system, the compatible polymer swells by absorbing some of the oily fraction of the bitumen to form a polymer phase distinct from the residual bitumen phase comprising the heavy fraction of the binder.

Many researchers have attempted different way to remove and improve the compatibility and stability of PMB such as sulphur vulcanization (addition of crosslinking agent), saturation, adding antioxidants, using hydrophobic clay minerals, functionalization (adding functional group) and adding reactive polymers (Nassar et al. 2012). These solutions do overcome some disadvantages of PMB, but most cause some new problems (Zhu et al. 2014). However, the addition of these compatibilizing agents may increase the production of PMB and thus limiting its application. In addition, Becker et al. (2001) reported that the stability of PMB can be improved if the acid is added to the bitumen prior the polymer. The phase separation or incompatibility can be obtained by several approaches, including phase compatibility, rheological changes and softening point variation.

2.9 Previous Studies on The Additive and Pre-treatment of PMB

Ait-Kadi et al. (1996) and Pérez-Lepe et al. (2003) used steric stabilization by copolymer to avoid polymer modified bitumen from phase separation. In their study, the base polymer used is a HDPE and the copolymer is an ethylene-propylene-diene monomer (EPDM). They concluded that the addition of copolymer into polymer modified bitumen results in more stable dispersion of base polymer and remains well-dispersed after more than 48 hours under a storage temperature of 160^{o}C (Ait-Kadi et al. 1996; Pérez-Lepe et al. 2003). In 2006, Perez et al. investigated the stability of different PMB at high temperature (180^{o}C) and they added reactive polymers

namely ethylene-glycidil methacrylate-acrylate (EGA) as bitumen additives to avoid phase separation problem. From the results, they concluded that the use of reactive polymers to modified bitumen could produce more stable binders.

Polacco et al. (2005) experimented by adding three types of copolymers of polyethylene namely acrylic acid (AA), glycidylmethacrylate (GMA) and reactive ethylene terpolymer (RET) into the PMB for improving the compatibility of PE-based polymer with bitumen. From the tests, they found out that no storage stability was achieved for all the PMB, even for the PMB incorporate with copolymers (Polacco et al. 2005).

In Casey et al. (2008), national authorities of Ireland specify that PMB only can be used in road surface if the minimum softening point of the PMB is 60^{o}C with penetration values less than 100. Therefore, they introduced two chemical additives namely Diethylnetriamine (DETA) and Polyphosphoric Acid (PPA) into the blends in order to meet the performance required. It was found that, the introduction of PPA into the blends showed that all binders exhibited more viscous at low temperature and satisfies the national requirement while no significant effect was observed when adding DETA to the binders (Casey et al. 2008).

Due to its incompatibility with bitumen, (Behl et al. 2014) in their study treated the waste PVC with a chemical modifier prior blending with bitumen in order to make a homogeneous blend. Besides that, Al-Hadidy and Tan (2008) utilized pyrolysis Polypropylene (PP) for producing a homogenous and storage stable PMB. Pyrolysis process is a process of breaking down of structure such as reduction of molecular weight (Billmeyer 1971). Polymer that having high molecular weight could reduce the compatibility of PMB (Becker et al. 2001).

Recently, Yu et al. (2015) conducted a study of adding additives which is organic montmorillonite (OMt) into the PMB for enhancing the storage stability. The 4% (by weight of base bitumen) of waste packaging polyethylene (WPE) was used as a control sample and then, the percentages of OMt added were varies namely 0.3, 0.6, 0.9 and 1.2% of bitumen weight. The results showed that the storage stability of PMB containing lower OMt had a significant improvement in storage stability but conversely occur to the higher content of OMt in PMB. WPE modified bitumens comprising high content of OMt tends to produce the OMt layers that would combine together during the shearing process and finally yielded agglomeration of OMt but when the OMt content is low, the layers tend to exfoliate during shearing and thus, no agglomerate generated. Better polymer dispersion occurred during high temperature storage of WPE/OMt modified bitumen because the exfoliated layers inhibited the floatation of the polymer (Yu et al. 2015). They found out that the exfoliation of OMt is the vital point to improve the storage stability of PMB. Besides that, the addition of OMt showed that increased in ductility and viscosity of PMB. In terms of its rheological properties, superior performance at high temperature was demonstrated by the WPE/OMt modified bitumens due to the enhancement of the cohesion of the modified bitumens containing two modifiers.

2.10 Modification Procedure

Chemical compatibility and modification processes, such as type of mixing device, time and temperature, are important to obtain the desired properties and stable compositions (Pérez-Lepe et al. 2003; Anjan Kumar & Veeraragavan 2011; Nassar et al. 2012) for improving the performance of modified bitumen (Fang et al. 2014b). Therefore, finding the optimal shear rate, the modification temperature and the

mixing time is essential to ensure optimum polymer/bitumen compatibility. The blending process is influenced by many factors as followed (Becker et al. 2001; Kalantar et al. 2012):

1. **Nature of the polymer**
    - Polymer that is high in molecular weight would need long time to be blended with bitumen and vice versa.
2. **Physical form of the polymer**
    - Smaller particle size has larger surface area per unit mass of polymer. Thus, the swelling of the polymer is easier and the penetration of the bitumen is facilitated. It means more rapid dissolution is completed. Powdered polymers will therefore disperse and dissolve more rapidly than porous pellets.
3. **Nature and grade of bitumen**
    - Bitumen with low viscosity can pre-disperse the polymer in itself and speed the penetration and swelling of the polymer particles. A low bitumen viscosity at the blending temperature can also improve the disintegration of the polymer at the mill.
4. **Type of mixing equipment**
    - There are two main methods for mixing the bitumen with polymer, namely, high shear and low shear mixing. Low shear mixer is a simple mixing tank with a paddle stirrer. It can be used to mix the bitumen with powdered modifier. Mixing process is limited to the swelling and dissolving the bitumen with polymer. The temperature is fixed during the mixing. High shear mixer reduces the polymer particles size by mechanical and hydrodynamic shear.

The temperature will increase during the mixing in order to dissolve polymer into the bitumen and make a homogenous blend.

5. Time-temperature profile during mixing

- The ideal mixing process should be undertaken at the lowest possible temperature for the shortest possible time, corresponding to the complete incorporation of the polymer into the bitumen both from an economic standpoint and to minimize any thermal effect on the polymer. Structure and properties of PMB is a function of blending conditions. It means the longer the mixing time is, the finer the microstructure will be and the higher the temperature is, the more rapidly this process is done.

- Many studies have been conducted in obtaining optimum blending parameters of PMB. In order to optimize the blending parameters, Al-Dubabe et al. (1998) utilized softening point and visual inspection using optical microscope for monitoring the homogeneity of polymer-bitumen blend for HDPE, PS, PP, LDPE, SBS, Polybelt and Crumb rubber tires (CRT). The homogeneity of PMB was evaluated by measuring the softening point and inspecting visually using microscope of the PMB blend at 20 minutes interval from the beginning of the blending operation. The optimum blending time (as shown in Figure 2.7) were obtained when no significant increase in softening point value at the particular blending temperature.

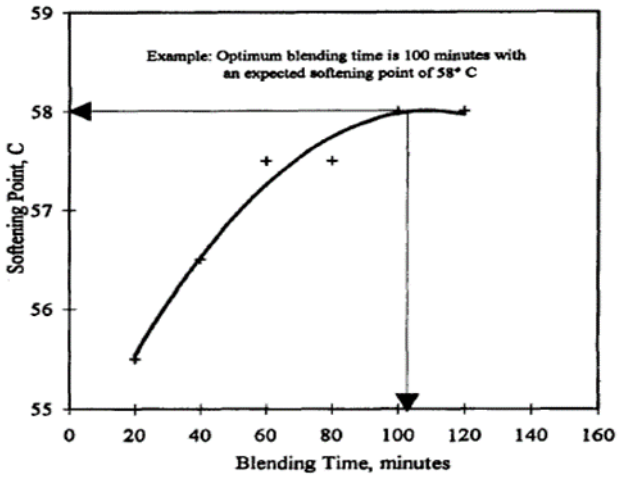


Figure 2.7: Determination of optimum blending time of PMB using softening point (Al-Dubabe et al. 1998)

Pérez-Lepe et al. (2003) investigated the influence of mixing process conditions on the rheological properties for unmodified and modified bitumen containing polymers and their asphalt mixtures. The modification of 5% polymer has been prepared by using two different mixers namely rotor-stator mixer at speed of 8200 rpm and stirred tank device at 1200 rpm which indicate a mixing device for pilot plant scale and laboratory scale, respectively. They concluded that in order to make the polymer stable and dispersed in bitumen, high energy mixing process is required although the changes of rheological properties of binder occurred due to the oxidation process. The polymer nature and blending process were vital to improve the mechanical properties of the modified bitumen (Pérez-Lepe et al. 2003).

Wahhab and Abaker (2004) study, they used melting temperature to identify the polymer concentrations, blending times and blending temperature. The polymers were rejected if having a melting temperature more than 180^{o}C and the blending temperature of the polymers were equal to the melting temperature plus 5^{o}C for ensuring good dispersion of polymers in the bitumen. They used the parameter,

G*/sin δ at 76^{o}C at interval of 30 minutes during the blending process to determine the optimum blending time and the maximum blending time was assumed to be two hours (Wahhab & Abaker 2004).

Habib et al. (2011) claimed that instead of solvating power of base bitumen and molecular structure of polymer used, the viscoelastic behavior of PMB also depend on the concentration of polymer, mixing temperature and mixing technique. They added that incorporate polymer in the powder form into bitumen was easy to produce a homogenous blend due to the linear decrease in penetration, increase in softening point and viscosity. The linearity of those results indicated that the continuous formation of stable internal structure of PMB (Habib et al. 2011). In the study, the mixer speed was maintained at 120 rpm with temperature was kept between 160^{o}C and 170^{o}C. In order to produce homogeneous mixture, the mixing time was continued for 1 hour.

In 2014 , Fang et al. studied the effect of preparation process of PMB on the stability. Different approaches of modification process have been done and the general procedures begin with 500 grams of base bitumen were heated at 190^{o}C at shear speed of 3750 rpm, then 20 grams of polymer was added into the base bitumen over 5 minutes and the mixture was sheared for 30 minutes. Other modifications were prepared by changing its blending time and blending temperature (190^{o}C for 1, 1.5 and 2 hours, 170^{o}C and 150^{o}C for shear time of 0.5, 1 1.5 and 2 hours). They found out that the optimum preparations conditions of PMB were prepared at shear rate of 3750 rpm with temperature of 150^{o}C for 1.5 hours. Samples prepared using these optimum conditions were found to exhibit good thermal storage stability, decreased sensitivity to temperature and the stability of bitumen was improved after

modification, while the preparation process had little effect on the thermal properties of the modified bitumen (Fang et al. 2014b).

Recently, Yu et al. (2015) used high speed shearing method for preparing the modified bitumen. Initially, they heated the 500g of base bitumen until it became fluid and then, the modifying agents were added individually into the container that sheared 3750 rpm in speed at temperature of 150^{o}C for 1.5 hours. For ensuring that the additives were fully swelling, the mixtures sat quiescent for 10 minutes after being sheared every 30 minutes of blending (Yu et al. 2015).

2.10.1 Blending Mechanism (Al-Dubabe et al. 1998)

A difficult operation has been identified for blending bitumen with high molecular weight materials particularly polymers because these two materials are heterogeneous and forced to form a two-phase system. The polymer must not dissolve but dispersed in the bitumen. Hence, a high shear rate of mixer which is normally more than 2500 rpm was required and recommended to perform the blending. Collins (1995) stated that for high shear mixing a Silverson L4R mixer operating at 4000 rpm was used while for low shear mixing an impeller low shear mixer operating at 1000 rpm was used. Fang et al. (2008) and Fang et al. (2014a) found that the modification with high speed shearing will disperse the polymer uniformly in the bitumen but turned out to be unstable during its storage at high temperature (Garcia-Morales et al. 2007).

Nevertheless, some polymer does not require high shear rate mixer such as SBS to well disperse in the bitumen although the time of blending can be reduced when using high speed. In fact, a high shear mixing needs a similar viscosity of the two components to be efficient, and this is the main problem in all PMBs production

where the bitumen has a very low viscosity in comparison to the polymer (Polacco et al. 2005).

2.10.2 Blending Time (Al-Dubabe et al. 1998)

The blending time relies extraordinarily on the mixing shear rate and mixing temperature. Long blending time could create economical, technical and practical effects and thus, minimum blending time was needed. Blending polymer into bitumen at high temperature for several hours may undergo changing in rheological properties and ageing phenomena which contribute to the deterioration of the pavement (Pérez-Lepe et al. 2003).

2.10.3 Blending Temperature (Al-Dubabe et al. 1998)

The molecular weight of the polymer has significant effect and highly depends on the blending temperature. High temperature of blending is needed for high molecular weight polymers such as HDPE while lower molecular weight such as SBS required low temperature blending. At a set temperature, the polymer particles' physical properties in bitumen would also affect the compatibility, and finally influence the modification effect of the bitumen (Fang et al. 2014b). Mixing temperature more than 190^{o}C should be avoided during mixing SBS polymer to prevent thermal effect occur (Brûlé et al. 1988).

2.11 The Ideal Quantity of Polymer to Use

Generally, the properties of PMB depend on the concentrations and the type of polymer used. In the performance of the entire pavement structure, the quality and quantity of bitumen play a significant role (Al-Dubabe et al. 1998). Although the percentage of the bituminous binder is relatively small, the bituminous binder

influences pavement performance more than the aggregate because environmental factors, such as heat and sun radiation, so affect bituminous binder more than aggregate (Polacco et al. 2005). The optimum amount of polymer to be used as a modifier in bitumen may depend on the bitumen and polymer sources (Chen et al. 2003). Polacco et al. (2004) stated that the amount of polymer added normally is about 4-6% by bitumen mass.

The rheological and engineering properties of the bitumen and asphalt concrete mixtures can be improved by the addition of small quantities of polymers from 3% to 8% by weight of bitumen (Al-Hadidy & Tan 2008). Despite of enhancing the binder properties, the addition of polymer by 2-6% of bitumen weight can reduce the maintenance costs although the production costs related to the mixing and storage may increase (Polacco et al. 2005).

Higher quantities of modifier (6-30% by weight) tend to rapidly cool at room temperature after mixing procedure and yielding a pseudo-solid material that is unstable from a thermodynamic point of view (Polacco et al. 2005). The addition of polymer in higher concentration is considered to be economically less viable and also may cause other problems related to the material properties (Polacco et al. 2004; Al-Hadidy & Yi-Qiu 2009b; Al-Hadidy & Yi-Qiu 2009c; Attaelmanan et al. 2011). Earnest (2015) found that the blending of 15% polymer with bitumen was difficult to obtain consistent DSR and viscosity result and thus the blends with this percentage were discarded. The amount of polymer to be used in the blend should be adjusted for getting a proper value of viscosity at the temperatures involved in bitumen application and compaction (Garcia-Morales et al. 2006). In Al-Dubabe et al. (1998) study, they used three approaches to identify the potential polymers in terms of: 1)

improvement in the rheological and performance properties of binders, 2) storage stability of PMB, and 3) cost effectiveness of the polymer modification. These approaches were evaluated to identify the effectiveness of the polymer used as modifier in improving the rheological properties of modified bitumen and satisfy the performance requirement of the Gulf countries.

Ait-Kadi et al. (1996) modified bitumen with 1, 3 and 5% of HDPE and HDPE/EPDM with composition of HDPE/EPDM was 90/10. After undergoing several tests such as ageing, conventional test and morphology test, it was found that polymer modified bitumen containing 1% of HDPE/EPDM showed the most promising results compared to other percentages and HDPE modified bitumen.

Wahhab and Abaker (2004) optimized the polymer content by evaluating the improvement of performance grade for different percentage of the polymer modified bitumen blends on Superpave binder performance. Results showed that 3% of polymer content in bitumen significantly improves the permanent deformation and fatigue life of asphalt mixtures.

In Al-Hadidy (2006), the polypropylene (PP) used was subjected to the pyrolysis (thermal degradation) process. Pyrolysis process is a process of breaking down of structure such as reduction of molecular weight (Billmeyer 1971). Then, the PP was grinding into the powder form and mixed with bitumen at different percentages of PP (1, 2, 3, 4, 6 and 8%). The PMB were blended at temperature of 150±5^{o}C for 7 minutes. He found out that pyrolysis PP and bitumen were compatible to extent. Later on, in 2008, Al-Hadidy and Tan used same material as Al-Hadidy (2006) used to modify bitumen for producing a homogenous and storage stable PMB. The blending process was performed for various percentages of pyrolysis PP (1, 3, 5,

7, 9 and 11%) at temperature of 160^{o}C for 5 minutes with low speed of stirring. Finally, the results showed that the optimum amount of pyrolysis PP was 5% for producing high performance and durability of pavement. A year later,Al-Hadidy and Yi-Qiu (2009c) studied the same material and blending process to modify the bitumen but different proportion of modifier (1, 3, 5 and 7% of bitumen weight). Similar optimum polymer content with the previous study has been obtained for better road surfaces.

In 2008, Casey et al. reported that blends that contain 6% of HDPE polymer were found to be fully saturated and therefore the consistency of the binder was less desirable. Consequently, a rough skin on the binder will be formed due to the loss of the glossy appearance associated with bitumen on the surface of these blends. Initially, the concentration of polymer that they used ranging from 2% to 5% by mass of bitumen and finally, they concluded that 4% of polymer was the most promising blended for improving binder performance.

In 2009, Al-Hadidy and Tan (Al-Hadidy & Tan 2009a; Al-Hadidy & Yi-Qiu 2009b) discussed the potential use of pyrolysis low density polyethylene (LDPE) as a modifier in hot mix asphalt mixtures. In the study, they mixed bitumen with different percentages of LDPE (powder form) 2, 4, 6 and 8% for 5 minutes at temperature of 160±5^{o}C at speed of 1750 rpm. They found out that the content of pyrolysis LDPE beyond 6% showed decreased in stability and increased in flow. They concluded that the used of 6% pyrolysis LDPE as modifier improved the performance of the mix.

Attaelmanan et al. (2011) studied the possibility of using HDPE as polymer additives to bitumen. They used different percentages of HDPE which are 1, 3, 5, and

7% in the pallet form and found that flexible pavement with high performance, durability and more economic can be obtained with 5% of HDPE.

Studied conducted by Habib et al. (2011) found that the formation of thermodynamically stable structure which may offer rutting resistance could be attained only when the addition of polymer concentration was kept below 3%.

Nassar et al. (2012) reported that the most promising concentration of PS as modifier in bitumen for improving workability and enhancing the resistance to deformation of asphalt mixture were recorded by 5% by weight of bitumen.

Sadeque and Patil (2013) stated that it is necessary to decide optimum dose of these waste polymer. Based on the study of waste LDPE and PP in bitumen, there was a significant improvement in the physical properties of the modified bitumen compared with those of neat bitumen and by considering the minimum ductility requirement criteria, they found that the optimum polymer content is about 4 to 5%.

Köfteci et al. (2014) investigated the performance of polymer modified bitumen based PVC in the powder form. The pure bitumen with penetration grade 160/220 was mixed with the modifier content of 1, 3, and 5% of bitumen weight to obtain PMB. It was found that waste polymer with 3 and 5% improved the performance of PMB at high temperature while the amount 5% of PVC improved the performance of binder at low temperature. Based on the experimental results, they concluded that the optimum amount of waste PVC to be used for obtaining best result was 3%.

So, it can be seen that from previous studies, the researchers obtained different optimum amount of polymer and this depends on the type of polymer used and the properties of binder that have been improved. Optimum amount of polymer

was needed for producing more economic pavement with better performance, long-term durability and thus minimizing maintenance cost.

2.12 Types of Polymer

The polymer can be classified in two categories, elastomer and plastomer (Wahhab & Abaker 2004; Nassar et al. 2011)

2.12.1 Elastomer

In the elastomer structure, it has a flexible backbone (like rubber) and large side-chain. Examples of elastomer are natural rubber, crumb rubber and the most popular elastomers as bitumen modifiers are styrene–butadiene–styrene (SBS) and styrene–isoprene–styrene (SIS). Incorporate bitumen with elastomer is able to confer good elastic properties on the modified bitumen (Al-Hadidy & Yi-Qiu 2009b; Al-Hadidy & Yi-Qiu 2009c) by stretching and recovering their original shape because of its high elastic response characteristic (Airey 2002; Airey 2004; Awwad & Shbeeb 2007; Cardone et al. 2014). Due to its rubber and polymer resin properties, elastomer exhibit excellent modification properties in both low and high temperature (Yu et al. 2015). However, unsaturated elastomeric polymers are quite expensive and subjected to degradation when exposed to atmospheric agents and mechanical stress (Polacco et al. 2005).

2.12.1.1 Previous Studies of Elastomer as a Modifier

Table 2.4: Previous Studies of Elastomer as a Modifier

Researcher and Year	Base Binder (pen. grade)	RWP Content (wt. % based on bitumen)	Blending Procedure	Conclusions
Lu and Isacsson (1998)	• Four sources: - Venezuela - Mexico - Saudi Arabia - Russia	• 3, 6, and 9% SBS: - Kraton D-1101 - Kraton D-1184	• Not stated	• that the SBS-modified bitumen demonstrated better rheological properties compared to the base binders • Due to ageing, the SBS polymer is degraded and oxidation occurred in the bitumen. • The SBS polymer may act as an antioxidant to formation of sulphoxide as evidence of lower IR absorbance of the sulphoxide group.
Wen et al. (2002)	• AH-90	• SBS 4303 • SBS 1301 • SBS 1401 • Additive: Sulphur	• High speed shear mixer at temperature of 160^{o}C for 1 hour	• the high temperature storage stability of the modified binder and the compatibility was improved by the addition of the sulphur.

			• Another 60 minutes after adding sulphur	• the addition of sulphur to SBS-modified bitumen gave a better effect on its rheological properties in terms of high temperature performance and temperature susceptibility in which the improvement was more pronounced as the sulfur levels increased.
Airey (2003)	• Two sources: - Venezuela - Russia	• SBS: - 3, 5 and 7%	• Prepared with a Silverson high shear laboratory mill at temperatures between 170 and 185^{o}C until steady state condition achieved	• The viscoelastic properties of these bitumens were improved by increasing its complex modulus and elastic response • Ageing results showed that the viscous response of higher polymer content in base bitumens is increased might due to the degradation of the SBS copolymer.
Kök and Çolak (2011)	• B 160-220	• 2 to 6% with 1% increment SBS: - Kraton D-1101 • Crumb rubber: - 3 to 15% with a 3% increment	• Laboratory-scale mixing device with a four-blade impeller (IKA) at a temperature of 180 °C for one hour at a rotation	• Both modifications improved the temperature susceptibility but more significant for the SBS-modified bitumen. • Based on the rheological and mechanical result of bitumen and asphalt mixture test, higher content of

			speed of 1000 rpm.	crumb rubber is required to have the same performance as the lower amount of SBS.
Liu et al. (2014)	• SBS PMB (Caribit Plus 85)			• A lower viscosity was exhibited by the lab-aged after 9 days in comparison with the 22 years field-aged binder and the dynamic response of the aged binder with 5-days lab shows a comparable result with the field ageing • In terms of chemical properties, the formation of asphaltenes in the lab ageing binders was more compared to the field-aged binder.
Swamy et al. (2017)	• VG 30 asphalt binder	• SBS: - Kraton D-0243K - Kraton D-0243KT • HDPEHP • The overall polymer content in asphalt binder was restricted to 6% by weight of binder. Within 6% polymer, percentage of HDPEHP was varied between 0% and 2%.	• SBS was blended at high shear of 3000 rpm for 2.5 hours at 180^{o}C • Later, HDPEDP was added for additional 30 minutes	• The blend containing SBS and HDPEHP gave positive effect on the rheological properties improvements with the optimal percentage of additive for modification is 4.5% SBS and 1.5% HDPEHP. • This blend exhibited superior performance at higher temperatures as evidence of lower phase angle and higher complex modulus at all conditions of binder.

2.12.2 Plastomer

Plastomer or also known as thermoplastics offer an increase in the stiffness of binder at high temperature and loading but unable to increase the elastic component of the binder. There examples of plastomer including high-density polyethylene (HDPE), low-density polyethylene (LDPE), linear low-density polyethylene (LLDPE), Polyvinyl Acetate (PVA) and so on. At normal service temperature, this polymer provides an increase in viscosity and stiffness of bitumen (Awwad & Shbeeb 2007). When subjected to the heat, thermoplastics polymer normally can be shaped into the new model. PMB that cooperate with plastomer can improve rigidity and reduce deformations under loads (Stastna et al. 2003; Al-Hadidy & Yi-Qiu 2009b; Al-Hadidy & Yi-Qiu 2009c) because it formed from a tough, rigid and three-dimensional network (Airey 2002; Airey 2004; Cardone et al. 2014). Unfortunately, these improvements could be made when the concentration of plastomer was less than 1% by weight of bitumen (Habib et al. 2011). Besides that, plastomer could increase the viscosity when blending with bitumen event at ambient temperature and thus increase the stiffness at service temperature but no significant changes in elastic behaviour observed (Stastna et al. 2003; Habib et al. 2011).

2.12.2.1 Previous Studies of Plastomer as a Modifier

2.12.2.1.1 Polyethylene Terephthalate (PET)

Table 2.5: Previous Studies of PET as a Modifier

Researcher and Year	Base Binder (pen. grade)	RWP Content (wt. % based on bitumen)	Blending Procedure	Conclusions
Mahrez and Karim (2010)	• 80/100	• 2, 4, 6 and 8%	• Wet process – High speed shear mixer at temperature of 130°C for 3 minutes.	• Addition of PET showed an improvement in the bitumen properties as the PET content increase. • The recycled PET modified bitumen has higher softening point and viscosity, lower penetration value and better viscoelastic properties such as higher complex modulus and lower phase angle compared to unmodified bitumen. • The viscosity value at 135°C of recycled PET modified bitumen were lower than 3kPas. • Recycled PET modified bitumen improves its rutting resistance compared to unmodified bitumen.

Kalantar et al. (2010)	• 80/100	– 2, 4, 6, 8 and 10%	• Dry process – Mixed manually for 2 minutes at temperature of 160^{o}C	• PET modified binder had lower penetration value and higher softening point. • Viscosity results were increased but the values are less than 3 Pas and therefore satisfy the ASTM criterion for binder workability • PET modified binder is stiffer and exhibit more elastic at high temperature • The addition of PET in bitumen improved the performance of the binder in high temperature area.
Modarres and Hamedi (2014)	• 60/70 from Tehran oil refinery	• PET: – 2, 4, 6, 8 and 10 • SBS: – 4, 5 and 6	• PET – Dry process • SBS: – Wet process – High shear mixer	• Addition of more than 2% PET reduced the resilient modulus at temperature of 5 and 20^{o}C. However, at all PET contents the resilient modulus quantities were in acceptable limit. • PET improved the fatigue behaviour and addition of the PET content up to 10% had beneficial effects on the fatigue response of modified mixes.

				• At 20°C PET and SBS had similar effects on the stiffness mixes but at 5°C, SBS reduce the mix stiffness which is ideal to improve the material flexibility at low temperature. • PET and SBS improved the fatigue response of mixes but SBS modified mixes showed better fatigue behaviour compared to PET at higher strain levels of 200 microstrain.
Mohammed and Hussein (2014)	• 40/50 from Baiji refinery	• 1, 2, 3, 4 and 5%	• Wet process – Blending at temperature of 155±5°C for about 40-50 minutes at speed of 2000 rpm.	• The addition of pyrolysis PET decreases the penetration, increase softening point, increase the penetration index which is better in hot climates. • PMB kept the ductility values at a minimum range of ASTM and SCRB specification of 100+ cm up to 4% of PET content. • the resistance of modified bitumen to the action of temperature and heat and temperature changes is increased

				• Compatibility can be better when the polymer is exposed to thermal degradation. • Good homogeneity and storage stability for PMB can obtained when adding 4% of PET.
Gürü et al. (2014)	• 45 from Turkey petroleum refinery	• 1, 2, 3, 5 and 10% • 1:2 for thin Liquid Polyol PET (TLPP) • 1:1 for Viscous Polyol PET (VPP) • Additives: – Glycol (Propylene glycol (PG)) – Catalyst (Titanium IV butoxide (TBT))	• Wet process – Blending at temperature of 120^{o}C for 10 minutes with a mechanical four-armed mixer rotating at 1300 rpm	• TLPP and VPP modifications reduced rutting resistance but offer significantly improved fatigue cracking resistance based on the DSR test. • They increased the stripping resistance of the asphalt mixture and enhanced the performance of bitumen at low temperatures. • At low VPP concentration showed an improvement in resistance of rutting. • At low bitumen content, TLPP modification improved the mixture stability. • Both TLPP and VPP modified bitumen were recommended to use in cold and humid regions.

Earnest (2015)	• PG 67-22 from Axeon, Savannah.	• 5, 10 and 15%	• Wet process – At mixing speeds of 3000 and 5000 rpm using Silverson high shear mixer and mixed for one and two hours at each mixing speed with temperature between 175 and 225^{o}C • Dry process	• PET did not adversely affect high temperature workability of the bitumen because all viscosities showed below the standard maximum value of 3Pas. • Addition of PET improved the high temperature performance of all bitumen. • The two hours mixing time produced more homogeneous blend. • PET did not contribute excessive volatiles to the mixture • The optimum content of PET is 10% whereby mixed at 3000 rpm for two hours and the 15% of PET was discarded because the excess PET particle in the blend made it difficult to obtain consistent DSR and RV results. • Dry process showed that PET improved stripping resistance, least susceptible to moisture damage, increase stiffness and recovery of the mixture.

				• The wet process with high shear rate speed provides better performance results than the dry process.
Kalantar et al. (2009)	• 80/100	• 2, 4, 6, 8 and 10%	• Wet process – Mixing PET with bitumen manually for about 2 minutes at 160^{o}C	• The mixture containing PET resulted in higher resistance to permanent deformation due to higher softening point. • Adding PET appears to decrease the consistency and increase the resistance of the material to temperature changes while the resistance to flow also increases.

2.12.2.1.2 High Density Polyethylene (HDPE)

Table 2.6: Previous Studies of HDPE as a Modifier

Researcher and Year	Base Binder (pen. grade)	RWP Content (wt. % based on bitumen)	Blending Procedure	Conclusions
Ait - Kadi et al. (1996)	• 150/200 from Ultramar company	• 1, 3 and 5% of HDPE and HDPE/EPDM (Blend compositions 90/10 (HDPE/EPDM)) • Additives: – Ethylene-Propylene-Diene terpolymer (EPDM)	• The polymer was mixed with bitumen at 170°C using a high shear speed stirrer rotating and then stored in small metallic containers at -4°C.	• HDPE blended with EPDM was found to impart enhanced overall properties of bitumen. • The 1% HDPE/EPDM modified bitumen presents the most promising results. • The presence of EPDM in the mixture found to stabilize the morphology before TFOT ageing.
Pérez-Lepe et al. (2003)	• 60/70 • 150/200	• Ethylene-propylene-diene monomer (EPDM), LDPE, HDPE and SBS – 5% with blend compositions 25/75, 50/50 and 75/25 (EPDM/PE)	• Laboratory scale: – 180°C at speed rate of 1200 rpm • Pilot plant scale: – Operates at 8200 rpm and temperature of 180°C	• The addition of copolymer has been proved to be an effective way in producing chemical stabilization of an insoluble base polymer into bitumen. • Compared to other modifiers and lab scale mixer, result from rotor-stator device on the HDPE and HDPE/EPDM modified bitumen proved that this

				pilot scale mixer with high energy mixing process can significantly improve the mechanical properties of the binder.
Wahhab and Abaker (2004)	• Not stated	• LLDPE, HDPE, LDPE, PP, MDPE and Styrenic Heavy Waste (SHW): – 3, 4 and 5%	• The blending temperature was made equal to melting temperature plus 5^{o}C for 2 hours at speed of 2500 rpm of 2500 rpm	• Mixtures with HDPE showed a significant increase in resilient modulus at low and high temperature compared to control mixture. • Modified bitumen showed a significant variation in their water susceptibility. • The average percentage loss in strength due to moisture damage was reduced effectively by HDPE. • Polymer modification showed an improvement in fatigue behaviour and rutting resistance at high and low temperatures.
Pérez‐Lepe et al. (2006)	• 60-70 provided by CESPA (Spain)	• 1, 2, 3, 4 and 5% • Additive: – A reactive terpolymer EGA (Ethyl-glycidil metha-acrylate (GMA)-butyl-acrylate)	• Prepared using a high-shear mixing device, at a pilot plant scale which is integrated a rotor-stator mixer with	• HDPE is partially swollen by some light aromatic components of the bitumen. Gross separations of the polymer phase occur after a short period of

			storage tank at a rotational speed of 8200 rpm and temperature 180°C.	storage at high temperature (above the melting point of PE). • The mechanism of the polymer-rich phase separation has been proved to be creaming, preceded by coalescence. • Owing to the incompatibility of the polymer with the bitumen, and the difference in densities, the PE-rich phase destabilizes and coarsens.
Pérez - Lepe et al. (2007)	• 60-70 provided by CESPA (Spain)	• SBS, LDPE, HDPE, EPDM/PE – 1, 2, 3 and 5% – 5% polymer and 2% EGA • Additives: – A reactive terpolymer EGA Ethyl-glycidil metha-acrylate (GMA)-butyl-acrylate – Ethylene-Propylene-Diene terpolymer (EPDM)	• Prepared using a high-shear mixing device, at a pilot plant scale which is integrated a rotor-stator mixer with storage tank at a rotational speed of 8200 rpm and temperature 180°C.	• HDPE containing binders show a remarkable enhancement of the mechanical properties compared to LDPE, EPDM and SBS. • Content of 3 and 5% of HDPE are able to form a polymer network that will enhance properties. • The use of EPDM/HDPE blend does not effectively hinder the separation of the PE from the bitumen matrix although a slight stabilization effect is apparent.

		– Blend compositions 25/75, 50/50 and 75/25 (EPDM/PE)		
Casey et al. (2008)	• 150 and 200 from Middle East	• LDPE, HDPE, and PP: • 2, 3, 4 and 5% • Additives: – Diethylenetriamine (DETA) – Polyphosphoric acid (PPA)	• Wet process – Firstly, the additive was mixed for 60 minutes and then recycled plastic was added and mix for a further 90 minutes at 160°C.	• HDPE and LDPE were found to be the most promise blend with a blend based on 4%. • Demonstrate enhanced performance compared to unmodified bitumen.
Attaelmanan et al. (2011)	• 80/100	• 1, 3, 5 and 7%	• The polymer was mixed at 170°C using a high shear speed stirrer rotating at a speed of 3000 rpm for 2 hours.	• The percentage loss of air and heat decrease with the addition of HDPE. • The ductility and uniformity test results indicated that HDPE and bitumen are compatible to an extent. • Addition of HDPE reduced the temperature susceptibility of bitumen and improved shear resistance in medium to high temperature.

				• The use HDPE in asphalt mixtures caused an increase in resilient modulus values at high temperature (25^{o}C). • Flexible pavement with high performance, durability and economic can be obtained with 5% HDPE.
Habib et al. (2011)	• 80/100 from Petronas refinery	• LLDPE, HDPE and PP – 0.5, 1, 1.5, 2, 2.5, 3 and 5%	• Mixing was done at speed of 120 rpm and temperature was kept between 160 and 170^{o}C for 1 hour.	• LLDPE and PP modified bitumen shoes least variation in penetration and softening point comparison to HDPE modified bitumen. • At lower concentration of the polymer in blend rapidly enhances the hardness of the PMB. • PP with linear structure added in powder form was easier to blend and produce homogeneous blend. • HDPE offer better resistance to rutting.

2.12.2.1.3 Polyvinyl Chloride (PVC)

Table 2.7: Previous Studies of PVC as a Modifier

Researcher and Year	Base Binder (pen. grade)	RWP Content (wt. % based on bitumen)	Blending Procedure	Conclusions
Köfteci et al. (2014)	• 160/220 from Turkish Petroleum Rafineries Corporation (TURPAS)	• 1, 3 and 5%	• The modifier was slowly added to the bitumen, and then mixed in the mixer at 500 rpm speed for about 15 minutes until it reached 180^{o}C. After reached the temperature, the mixer speed was raised to 1300 rpm and continued for another 60 minutes.	• Waste PVC modified bitumen able to improve the low temperature PG, and become more resistant to high temperature and heavy traffic condition • Modification of bitumen increases the hardening in the bitumen but in term of ageing, it did not exceed the limit values defined in the Superpave specification. • The modification of pure bitumen with waste PVC show better results with the optimum content of this waste was 3%.

Behl et al. (2014)	• 80/100 from Mathura refinery India	• 3 and 5%	• Mixing was performed by using a stirrer with the speed of 2000 rpm for 2 to 3 hours at 160^{o}C.	• The addition of PVC waste to the bitumen enhances both the binder's as well as the mix's properties. • The PMB shows better resistance to the permanent deformation and increases the values of indirect tensile strength to resist cracking. • The asphalt mixtures containing PMB showed increase in retained stability which indicated that the bond between the aggregate and the binder becomes stronger with the addition of polymer. • Addition of waste PVC reduced the rutting values of the asphalt mixture and increased the fatigue life.

2.12.2.1.4 Low Density Polyethylene (LDPE)

Table 2.8: Previous Studies of LDPE as a Modifier

Researcher and Year	Base Binder (pen. grade)	RWP Content (wt. % based on bitumen)	Blending Procedure	Conclusions
Polacco et al. (2005)	• 70/100	• LLDPE and LDPE – 2, 4, and 6% • Addatives: – Acrylic acid (AA) – Glycidylmethacrylate (GMA) – RET Copolymer	• When the asphalt temperature reached 180°C, a high shear mixer was dipped into the can and set to 4000 rpm. The polymer was added gradually (about 5 g/min) while keeping the temperature within the range of 180±1°C during the polymer addition and the subsequent 2 h of mixing. • Then the samples were stored at -20°C.	• The purpose of adding copolymers were to improve the compatibility of polymer with bitumen but no significant improvement in storage stability was observed. • The LDPE modified bitumen showed a greater compatibility with the bitumen and enhanced the mechanical properties compared to other blends.

Al-Hadidy and Tan (2009a)	• 50/60 from Baiji oil refinery	• 2, 4, 6 and 8%	• LDPE was subjected to pyrolysis process. • The modification was made by using high shear stirrer rotating at speed of 1750 rpm for a period of 3-5 minutes at 160±5°C.	• SMA modified with LDPE may resist the permanent deformation forces, rutting and shoving. Therefore, it should be used on busy intersections or truck stops and parking lots where standing loads caused extended periods of such deformation.
Al-Hadidy and Yi-Qiu (2009b)	• 50/60 from Baiji oil refinery	– 2, 4, 6 and 8%	• LDPE was subjected to pyrolysis process. • The modification was made by using high shear stirrer rotating at speed of 1750 rpm for a period of 3-5 minutes at 160±5°C.	• The addition of LDPE in bitumen improved shear resistance in medium to high temperatures, enhanced the resistance to deformation, reduce the temperature susceptibility and compatible to an extent. • The LDPE modified asphalt mixtures may reduce the cracking potential of pavement at low temperatures; prevent bleeding phenomenon and does not weak when exposed to moisture. • The optimum pyrolysis LDPE is 6%.

Fang et al. (2014b)	• 90A from China Petroleum and Chemical Corporation	• 20 grams	• The steps: i. Heating bitumen to 190^{o}C ii. Adding modifier over 5 min with speed: 3750 rpm iii. Sample 1: – Then sheared for 30 min. – Temperature: 190^{o}C iv. Sample 2-4: – Time: 1, 1.5 & 2 h – Temperature: 190^{o}C v. Sample 5-8: – Time: 0.5, 1, 1.5 & 2 h – Temperature: 170^{o}C vi. Sample 9-12: – Time: 0.5, 1, 1.5 & 2 h – Temperature: 150^{o}C vii. For each process, modified bitumen	• The optimum preparation conditions were found to be; shear rate of 3750 rpm, a shear temperature of 150^{o}C with the shear time of 1.5 hours. • Samples prepared were found to exhibit good thermal storage stability, decreases the sensitivity to temperature and improved the stability after modification. • While the preparation process had little effect on the thermal properties of the modified bitumen.

			swelled for 10 min after each half hour of shearing.	
Sadeque and Patil (2013)	• 60/70 from Shell Corporation	2, 4, 6 and 8	• Waste LDPE and PP modified bitumen is prepared by heating bitumen at 200^{o}C and waste plastic mixed and stir for one hour.	• Addition of waste in bitumen showed better physical properties than neat bitumen and exhibited greater temperature susceptibility. • Higher percentage of modifier decreased the ductility of PMB and not suitable for modification. • The optimum waste polymer content is about 4 to 5%.
Yu et al. (2015)	• 90A from China Petroleum and Chemical Corporation	• LDPE and LLDPE (waste packaging polyethene, WPE): – 4% • Additive: – Organic montmorillonite OMt: – 0.3, 0.6, 0.9 and 1.2%	• After adding the modifying agents, the mixture was sheared for 1.5 hour with the speed of 3750 rpm at 150^{o}C. • Then, the mixture sat quiescent for 10 minutes after being sheared every 0.5 hour.	• The storage stability of WPE/OMt modified bitumen is significantly improved when the OMt content is low. • The WPE modifying agents demonstrates improvement at the high temperature pavement performance. • OMt is more influence in the toughness of modified bitumen than a single WPE dose.

				• The WPE/OMt modified bitumen enhanced the storage stability and low temperature properties.

2.12.2.1.5 Polypropylene (PP)

Table 2.9: Previous Studies of PP as a Modifier

Researcher and Year	Base Binder (pen. grade)	RWP Content (wt. % based on bitumen)	Blending Procedure	Conclusions
Al-Dubabe et al. (1998)	• Not stated	• PE, PS, PP,LLDPE, SBS and Crumb rubber tires (CRT): – 2, 4, 6 and 8%	• The modified bitumen was prepared with high shear rate which is 2500 rpm at temperature between 160 to 200^{o}C for 2 hours.	• Polymer obtaining from second phase can satisfy the performance requirement of the Gulf countries with a particular percentage (especially for PP was 1.5% by weight of the total blend) but most of the polymer exhibited poor storage stability except for CRT modifier • Continues blending during hot storage will minimize the polymer separations.

Al-Hadidy (2006)	• 40/50 from Baiji oil refinery	• 1, 2, 3, 4, 6 and 8%	• PP was subjected to thermal degradation (pyrolysis process). • The bitumen was mixed with PP for 7 minutes at temperature of 150±5°C	• PP modified bitumen showed an improved shear resistance in medium to high temperature, increased the resistance to deformation, improve binder performance viscosity and reduced the temperature susceptibility of bitumen. • The percentage of loss of air and heat decrease with the addition of PP • PP modified asphalt mixtures showed less susceptible to stripping phenomenon, slight improvement in the retention of bitumen coating and resist permanent deformation forces, rutting and shoving.
Al-Hadidy and Tan (2008)	• 50/60 from Baiji oil refinery	• PP, low-density linear polyethylene (LLDPE), styrene-butadiene-styrene (SBS) and crumb rubber tires (CRT):	• PP was subjected to pyrolysis process • The bitumen was mixed with PP for 5 minutes at	• PP modified bitumen showed an improved shear resistance in medium to high temperature, increased the resistance to deformation and reduced the temperature susceptibility of bitumen.

		– 1, 3, 5 and 7%	temperature of 160±5°C.	• In SMA mixture, the pyrolysis PP was effective in preventing excessive drain down of the mixtures, exceeded the indirect tensile strength requirement set by the SCDOT and tough when exposed to moisture. • The optimum PP content was 5%
Al-Hadidy and Yi-Qiu (2009c)	• 50/60 from Baiji oil refinery	• PP, low-density linear polyethylene (LLDPE), styrene-butadiene-styrene (SBS) and crumb rubber tires (CRT): – 1, 3, 5 and 7	• PP was subjected to pyrolysis process • The bitumen was mixed with PP for 5 minutes at temperature of 160±5°C.	• The surface thickness of asphalt pavement containing of pyrolysis PP modified bitumen reduced by 40% for almost the same service life of modified and unmodified pavements. • The pavement consisting of pyrolysis PP modified SMA as a surface layer is beneficial in reducing the construction materials

2.12.2.1.6 Polystyrene (PS)

Table 2.10: Previous Studies on PS as a Modifier

Researcher and Year	Base Binder (pen. grade)	RWP Content (wt. % based on bitumen)	Blending Procedure	Conclusions
Fang et al. (2008)	• 110# from petrochemical factory in LAN Zhou	• PS and EVA • 0/0, 0/3, 3/3, 4/3, 5/3 and 6/0 (PS/EVA)	• The modification bitumen prepared at temperature of 190°C, stirrer for 40 minutes and then sheared by the shearing machine for 1 hour. After that, the mixture was kept at temperature of 120°C for 30 minutes. • After fully swelling, the mixture was sheared again by high speed shear machine for 60 minutes.	• The combination of PS and EVA improved the low temperature ductility and enhanced the stability of the bitumen colloidal system. • The comprehensive performance of the modified bitumen by waste PS and EVA was improved and it may provide a new way to reuse recycled polymers for reducing cost and pollution

Sham (2011)	• 60/70	• PS: – 4, 6, 8 and 10	• The PS was softened at 180°C for 30 minutes. • The bitumen mixed with PS at 500 rpm and 180°C temperature using laboratory mechanical stirrer	• The PMB showed an improvement in term of deformation resistance in medium to high temperature. • However, the ductility of PMB was very low indicated that the binder become very brittle after modification. • The optimum PS content was 8%
Nassar et al. (2012)	• 60/70 from El-Nasser Petroleum Company Suez-Egypt	• PS: – 2, 3, 4, 5 and 6%	• The PMB were prepared using a high shear mixer at 180°C and a speed of 600 rpm for 2 hours.	• The PMB gave higher stability values, increase in air voids and slightly decrease in flow. • PMB showed compatibility between polymer and bitumen matrix. • The polymer modification was less susceptible to the temperature changes, increased the viscosity values at high temperature indicated that the PMB offer better in rutting resistance. • The strategy behind modifying bitumen is to depend on the base bitumen to provide good low temperature properties while depending on the

				modifier to provide good high temperature properties.
Fang et al. (2014a)	• 90#	• 2, 3, 4 and 5%	• The mixture was bitumen and PS was heated to 190^{o}C, then manually stirring for 30 minutes and followed by automatic stirring for 1.5 hour. • Then, the mixture was then cooled to 120^{o}C and kept for 30 minutes to disperse PS in bitumen.	• The PMB showed an improvement in rutting resistance and the viscoelasticity. • The viscosity of PMB was greater that base bitumen and more significant at low temperature.

Table 2.11 summaries the advantages and disadvantages in using each type of polymer to modify bitumen while Table 2.12 shows the brief results of tests that have been done for the modified bitumen and its asphalt mixture for each type of polymer.

Table 2.11: Characteristics of polymers used to modify bitumen (Murphy et al. 2000; Jin et al. 2002; Polacco et al. 2005; Glover 2007; Pérez-Lepe et al. 2007; Casey et al. 2008; Al-Hadidy & Tan 2009a; Mahrez & Karim 2010; Tapkın et al. 2010; Habib et al. 2011; Kalantar et al. 2012; Nassar et al. 2012; Costa et al. 2013; Behl et al. 2014; Gürü et al. 2014; Mohammed & Hussein 2014)

No	Polymer	Advantages	Disadvantages
1	Polyethylene Terephthalate	• High rutting resistance • Recommended to be used in hot climate region • Improved the resistance of fatigue cracking • Resist to low temperature cracking	• High melting point hindered the mixing
2	High Density Polyethylene	• Able to sustain the pavement demands in the high temperature region • More compatible compare to other polymer when blending with bitumen • Offer better resistance to rutting • Good performance according to storage stability • Higher resistance against rutting • Better adhesion between aggregate and binder • High thermal extension	• Low stiffness • Almost completely immiscible with asphalt due to non-polar nature • No elastic recovery • High polymer contents are required to achieve better properties
3	Polyvinyl Chloride	• Lower cracking PVC disposal • Increase stability • Reduce rutting problem • Increase fatigue life	• Acts mostly as filler • High melting point hinder the mixing

4	Low Density Polyethylene	• Improve the performance of road surface in both high and low temperature area • Better adhesion between aggregate and binder • High thermal extension	• Low stiffness • Almost completely immiscible with asphalt due to non-polar nature
5	Polypropylene	• Good chemical resistance • Good fatigue resistance • High thermal expansion • Widens the plasticity range and improves load resistance of binder • Improve Marshall quotient and Marshall stability value • Improve the mechanical and physical properties of asphalt mixture • Reduce deformation under traffic loads	• Oxidative degradation • High mould shrinkage • Low thermal fatigue cracking resistance • Separation problem • No improvement in elasticity or mechanical properties • Almost completely immiscible with asphalt due to non-polar nature • Low viscosity
6	Polystyrene	• Improved thermal stability • Good dispersion in the bitumen matrix • Reduced the temperature susceptibility • Good property for rutting resistance • High temperature stability	• Very difficult to disperse into bitumen even at high shear rate

Table 2.12: The list of tests of PMB which have been conducted

Tests	Recycled Polymer					
	PET	HDPE	PVC	LDPE	PP	PS
Bitumen Tests						
Penetration test (dmm)	⁄	⁄	⁄	⁄	⁄	⁄
Softening Point (°C)	⁄	⁄	⁄	⁄	⁄	⁄
Viscosity test	⁄	⁄	⁄	⁄	⁄	⁄
Frass Breaking Point test		⁄				
Ductility test	⁄	⁄		⁄	⁄	⁄
Durability test		⁄		⁄	⁄	
Compatibility test (Morphology)	⁄	⁄		⁄	⁄	⁄
Storage Stability test	⁄	⁄		⁄	⁄	⁄
Dynamic Shear Rheometer (DSR)	⁄	⁄	⁄	⁄		⁄
Bending Beam Rheometer (BBM)	⁄		⁄			
Age Hardening (TFOT or RTFOT)	⁄	⁄	⁄	⁄	⁄	⁄
Thermal Analysis				⁄		⁄
FT-IR	⁄					⁄
Differential Scanning Calorimeter (DSC)						⁄
Scanning Electron Microscope (SEM)	⁄					
Mixture Tests						
Stability (kN)	⁄	⁄	⁄		⁄	⁄

Flow (mm)	⁄	⁄			⁄	⁄
Stripping Test (Moisture susceptibility)	⁄	⁄	⁄	⁄	⁄	
Resilient Modulus		⁄				
Flexural Fatigue Test		⁄	⁄			
Indirect Tensile Strength (ITS)	⁄		⁄	⁄	⁄	
Compressive Strength				⁄		
Wheel Tracking			⁄			
Rutting	⁄	⁄				
Compressive Strength					⁄	

2.13 Summary

Generally, the idea of bitumen modification with polymer is to provide good low and high temperature properties of road pavement. Based on the literature review, the following conclusion was drawn:

1. The addition of polymers has been proven to be significantly improved the rheological properties of the modified binder and affects the performance of asphalt mixture.
2. Characteristics of bitumen and polymer in terms of its micromorphology and mechanical properties greatly influence the properties and compatibility of PMB. The obtained results may vary if bitumen and polymer from other sources is used although same modification procedure is applied.
3. Optimum modification procedures such as blending mechanism, time and temperatures are vital to obtain desired properties and stable compositions.

i. Blending mechanism:

- A difficult operation has been identified for blending bitumen with high molecular weight materials. High shear rate of mixer is required and recommended to perform the blending, but some polymer may only need slow rate mixer to well disperse in the bitumen and produce homogenous PMB.

ii. Blending time:

- It depends on the blending speed of mixer, temperature and polymer form. Long blending time could affect the rheological properties of the PMB. Powder form of polymer was easy to disperse in bitumen, thus short blending time is required. Most of the previous studies blend the mixture with maximum of 2 hours at high shear rate. However, low shear rate may increase the blending time to produce a homogenous blend if the polymer is pallet form.

iii. Blending temperature:

- The molecular weight of the polymer has significant effect and highly depends on the blending temperature. High molecular weight of polymer would need high temperature of blending and vice versa. Most of the studies used blending temperature as low as 150^{o}C. Blending polymer with bitumen at temperature more than 190^{o}C should be avoided for preventing thermal effect.

4. The properties of PMB depend on the concentrations and type of polymer used. Higher concentrations of polymers are considered to be economically less viable and also may cause other problems related to the material properties such as

unable to obtain the result of the test consistently. Based on the previous studies, the optimum of polymer to modify bitumen was in the range of 3-6%.

5. Various solutions in producing compatible polymer and bitumen blend have been done such as sulphur vulcanization (addition of crosslinking agent), functionalization (adding functional group), adding reactive polymers and pre-treatment of polymer (pyrolysis process). However, these approaches may not have a significant effect on compatibility and also increase the cost of the PMB production.
6. The use of waste polymer is being considered for bitumen modification due to the high cost of the virgin polymer.
7. Many studies have been conducted on PMB and reported that polymer modification exhibit some improvement on bitumen properties to some extent but various disadvantages also appeared which will limit its application. Therefore, the needs still exist to solve these problems for developing more effectives PMB and promote a further development.
8. The potential polymers to be used as a modifier can be identified in terms of improvement in the rheological and performance properties, storage stability, durability upon moisture damage and ageing conditions and its cost efficiency.

CHAPTER 3: **EXPERIMENTAL PROGRAMME**

3.1 Introduction

This chapter provides the details of the laboratory experimental work involved in this study. It begins with a description of the materials used and then followed by the experimental setups for binder tests and asphalt mixture tests. For each test, the data interpretation technique is discussed.

3.2 Material

3.2.1 Binder

In this study, the RWP-modified binders were produced with the addition of the RWP into the base binder. The base binder used was a 160/220 penetration grade bitumen (referred as BB). This binder type was chosen because soft bitumen have good compatibility between polymer and bitumen due to its lower asphaltenes content (Zhu et al. 2014). Then, after manufacturing the RWP-modified binder, the other two conventional binders is used for later comparison which are the 40/60 penetration grade bitumen (referred as control binder, CB) which represents the similar penetration grade of high modification (7% RWP-modified binders) of base binder and SBS-modified bitumen representing a ready-made polymer modified bitumen (PMB)Table 3.1 gives a summary of the properties of these binders.

Table 3.1: Fundamental Properties of the base binder

Binder	Penetration (dmm)	Softening point (°C)
Base Binder (BB)	181	37.4
Control Binder (CB)	47	50.4
SBS-modified Bitumen (SBS)	87	84.2

3.2.2 Recycled Waste Plastic (RWP)

In this study, the RWP used as modifiers were Polyethylene Terephthalate (PET), High Density Polyethylene (HDPE), Polyvinyl Chloride (PVC), Low Density Polyethylene (LDPE), Polypropylene (PP) and Polystyrene (PS). Those RWPs were obtained from a local recycler namely the B & J Parr Company located at Mansfield, Nottinghamshire. Figures 3.1 to 3.6 shows the image of the RWPs.

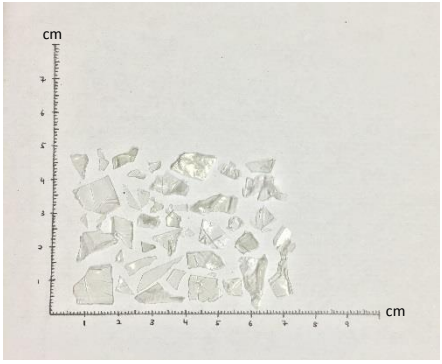


Figure 3.2: PET

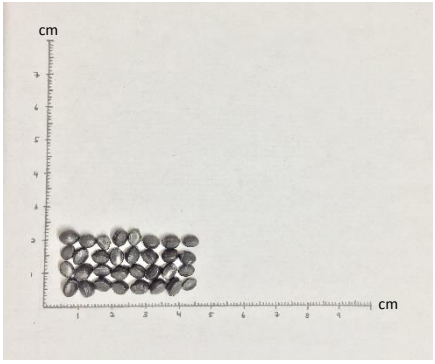


Figure 3.1: HDPE

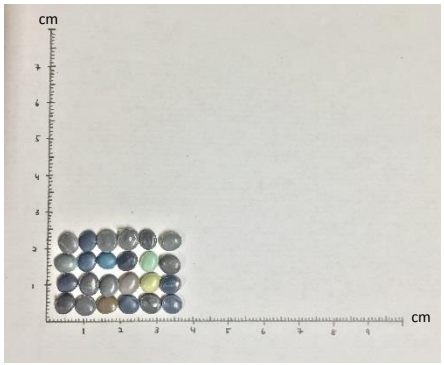


Figure 3.3: LDPE

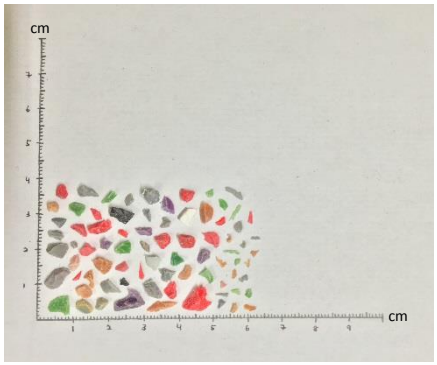


Figure 3.5: PVC

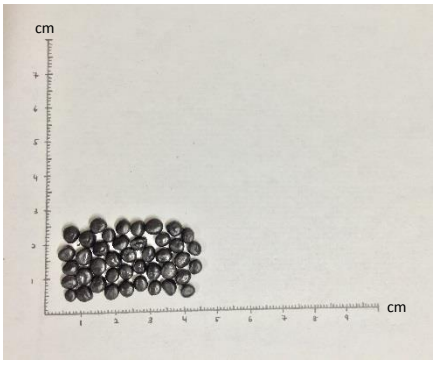


Figure 3.4: PS

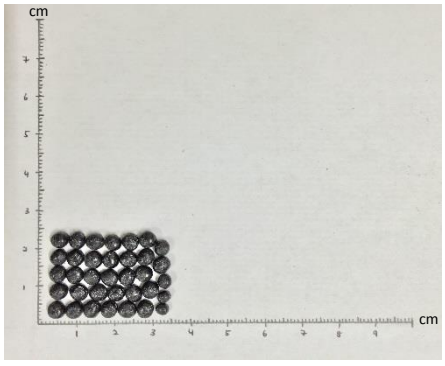


Figure 3.6: PP

3.2.2.1 Differential Scanning Calorimetry (DSC)

The Differential scanning calorimetry (DSC) is used to observe the behaviour of the sample in terms of heat capacity (Cp) when subjected to the change on the temperature in a controlled atmosphere. When heated or cooled, the changes in heat capacity of the known mass sample is recorded and tracked as changes in the heat flow. Then, the changes in structure (transitions) such as melts, glass transition and phase changes can be identified. Dalhat and Al-Abdul Wahhab (2015) employed DSC to determine the exact melting point of the RWP and then, the screening of RPWs that can be used to modify the base binder was done based on these thermal analysis results.

The configuration of the test is displayed in Figure 3.7. The test consists of two pans namely one pan is the sample pan and the other pan is the reference pan which is empty. Then, these two pans were placed on the top of a heater and this heater will heat these two pans at a specific heat flow. During the test, the temperature increased of these two pans should be at the same rate. When the heat is flowing, the sample was undergone structure changes and more heat is needed to perform the transition. In order to ensure that the heat is flowing at the same rate as the reference pan, the sample pan must be heated more and DSC will measure the heat needed for the sample pan. The output of the test is plotted in term of the heat absorbed by the sample against temperature (Figure 3.8).

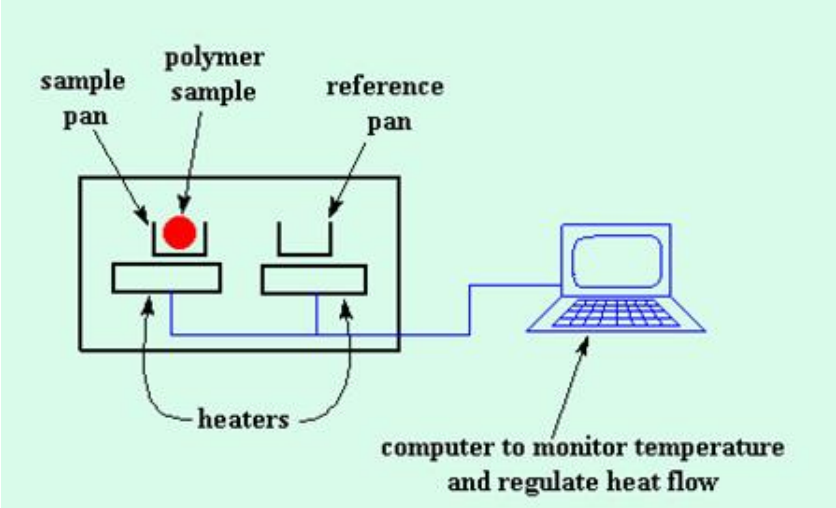


Figure 3.7: Configuration of DSC test

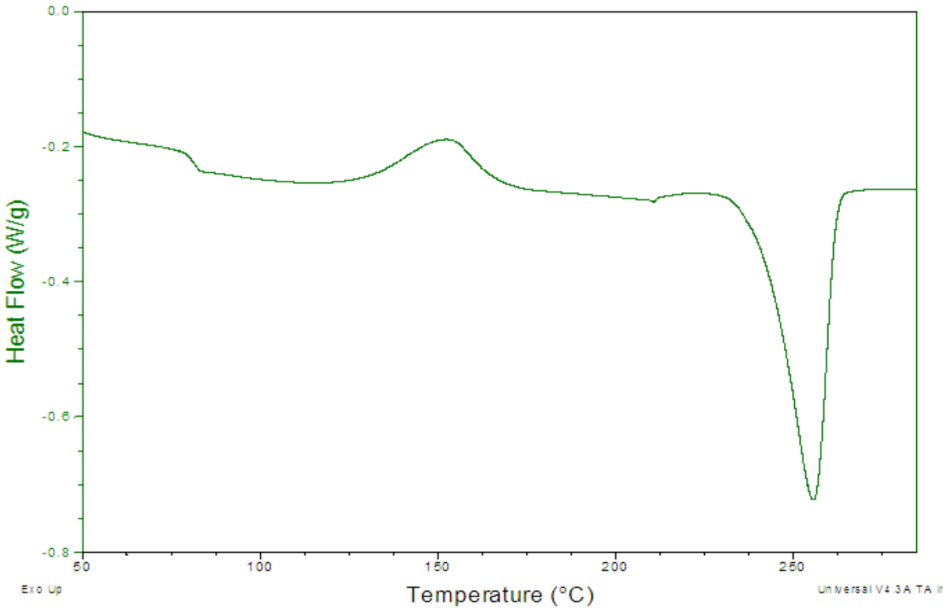


Figure 3.8: Typical DSC result

3.2.2.1.1 Testing Protocol

A sample about 10 mg was sealed in hermetic aluminium pan as depicted in Figure 3.9. Then, this sample was subjected to the thermal test by using TA Discovery DSC 2500 machine (Figure 3.10) for obtaining its melting temperature and the test has been carried out under a heating rate of 10^{o}C/min. The hermetic aluminium pan was

clean with ethanol before loading the sample into the pan and similar procedure was done for the reference pan.

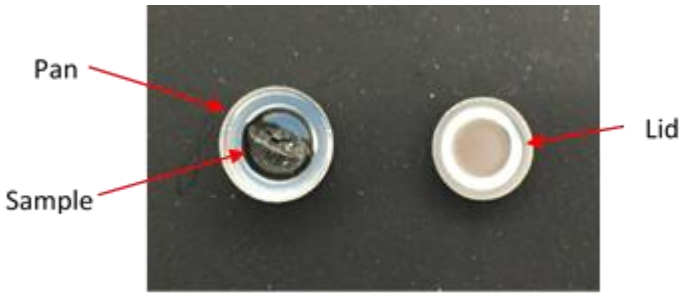


Figure 3.9: Sample in the hermatic aluminium pans

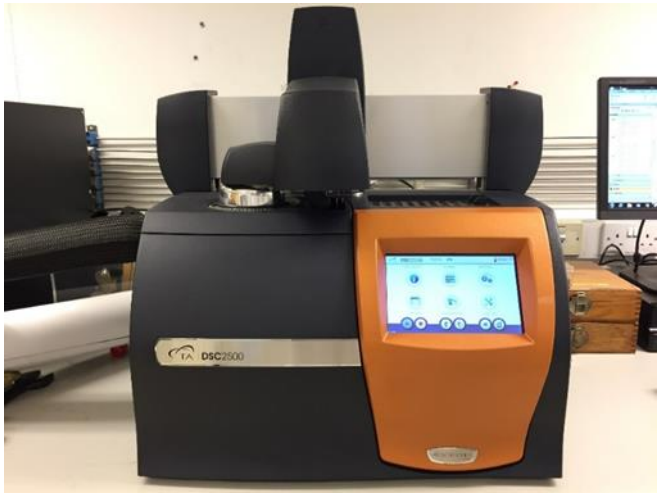


Figure 3.10: Differential Scanning Calorimetry (DSC) machine

3.3 Binder Tests

Moreno-Navarro et al. (2017) reported that there is a strong relationship between bitumen and the asphalt mixture properties. Thus, the evaluation of the binder properties from the binder tests is useful to predict the asphalt mixture performance. Moreover, these binder results could be used as a guideline for selecting binder in asphalt mixture and then, helps in saving time and materials(Jiménez del Barco Carrión 2017).

3.3.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared, known as FTIR is used to identify organic, polymeric and some cases inorganic compound. Lamontagne et al. (2001) and Permanyer et al. (2002) stated that this test is sensitive, convenient and able to give reliable information regarding chemical aliphaticity, aromaticity and oxygenation rate. In this study, FTIR was used to evaluate if ageing occurred during the blending of RWP within the base binder.

3.3.1.1 Principles

FTIR test was conducted using the infrared spectroscopy method. This method used infrared (IR) light to scan the sample and then an IR spectrum of absorption or emission of the sample is obtained in term of wavelength (μm) or more often wavenumber (cm^{-1}). The resulting wavenumber or wavelength indicate the presence of different functional (Bowers 2013) and often described as the 'fingerprint' of the bitumen (Airey 1997). The measurement of IR absorption is presented in the plot that consists of a spectrum with wavenumber (or wavelength) on the x-axis and absorption band on the y-axis (Burns 2014).

The relationship between wavenumber and wavelength can be represented as follow (Burns 2014):

$$wavenumber = \frac{1}{\lambda} \qquad \text{Equation 3.1}$$

Where:

λ = wavelength

The principle of the FTIR test is that the energy from the IR light source is transmitted through the bitumen with wavenumber of 400 cm^{-1} to 4000 cm^{-1}. This

energy causes bond in the molecules of the bitumen to vibrate at frequency which is depends on the chemical functional groups present in the bitumen.

Burns (2014) explained that when IR light is transmitted through a sample in a FTIR spectrometer, molecules within the bitumen will absorb the energy when the frequency of vibrational energy is matched with the IR radiation frequency. An IR spectrometer will measure the absorbed radiation amount at a particular wavenumber and displayed in IR spectrum as an absorption band. Then, a series of absorption bands at a specific wavenumber is recorded as transmittance (%) or absorbance against the wavenumber (Airey 1997).

3.3.1.2 FTIR Spectrum Calculation

The major functional groups for identifying the ageing properties of bitumen is the sulfoxide (S=O) group (wavenumber found at 1020 cm^{-1}) and the carbonyl group (wavenumber found at 1702 cm^{-1}). The measurement of these functional groups can be obtained by performing spectrometric indices calculation method that has been introduced by Lamontagne et al. (2001). The band areas measured from valley to valley is used to calculate the functional and structural indexes. Figure 3.11 shows the main FTIR bands used for the original bitumen. The band area ratio allows us to calculate several index types for obtaining the index of chemical composition of each component. These indices are calculated as follow:

1. Aromaticity index: $A_{1600}/\sum A$
2. Aliphatic index: $(A_{1460} + A_{1376})/\sum A$
3. Oxygenated functions:
    a. Carbonyl index: $A_{1700}/\sum A$

b. Sulphoxide index: $A_{1030}/\sum A$

The sum of the area represents:

$$A = A_{1700} + A_{1600} + A_{1460} + A_{1376} + A_{1030} + A_{864} + A_{814} + A_{743} + A_{724} + A_{(2953,2923,2862)}$$ Equation 3.2

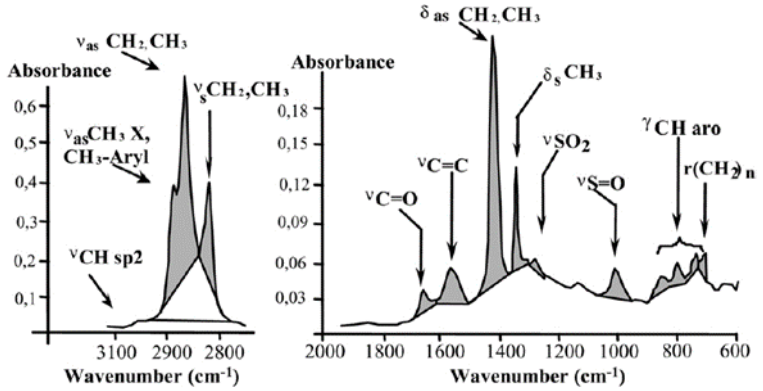


Figure 3.11: FTIR spectrum of a 4000-400 cm-1 original bitumen. Band areas measured from valley to valley.

3.3.1.3 Testing Protocol

The FTIR analysis of sample was carried out using the Bruker Tensor-27 FT-IR Spectrometer as shown in Figure 3.12. Around 0.5 g of binder is poured on the mould and leave it to be solid at ambient temperature before being tested in the machine. When the binder become solid, the sample is placed on the FTIR crystal as shown in Figure 3.13 (in the dashed circle) and before conducting each test, the ethanol was used to clean the crystal. Figure 3.14 displayed a sample that is ready to be tested. A total of 32 scans was conducted and the average of these scans is recorded and yield a FTIR spectrum as a result (Figure 3.15).



Figure 3.12: FTIR machine

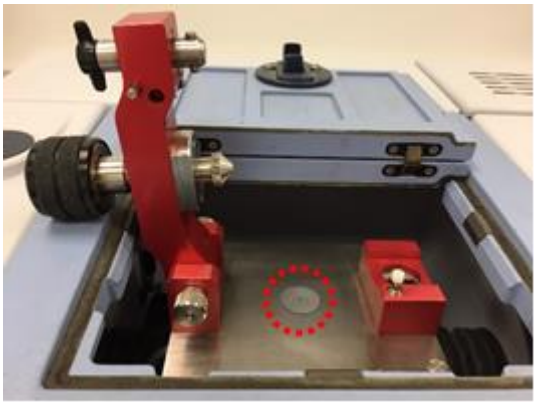


Figure 3.14: FTIR Crystal

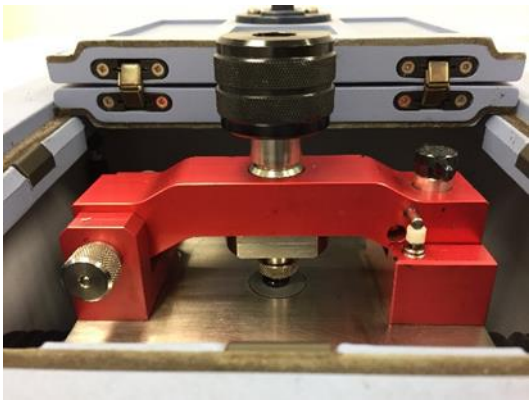


Figure 3.13: Sample configuration

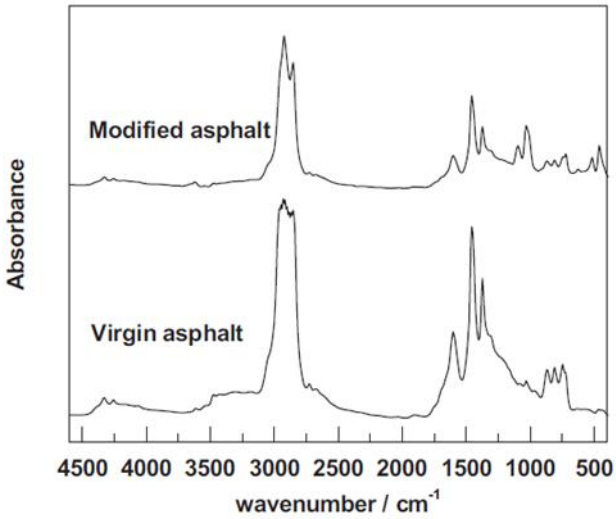


Figure 3.15: Typical result of FTIR spectrum (Fang et al. 2012)

3.3.2 Storage Stability Test

The stability of the RWP-modified binders during hot storage temperature storage stability test was conducted on RWP-modified binders using the toothpaste test according to BS 2000-517:2010. The Dynamic Shear Rheometer (DSR) was used to obtain the rheological properties of the binders. Then, the separation of RWP and base bitumen was evaluated by comparing the rheological property which is complex modulus (G^*) of top and bottom sections taken from the toothpaste tube.

3.3.2.1 Testing Protocol

The storage stability test was done by pouring the homogeneous RWP-modified bitumen into a tube or known as toothpaste tube to a height of 100 mm to 120mm. Then, the tubes were sealed and stored in the oven at 180^{o}C in a vertical position for 6, 24 and 72 hours. After that, the sample tubes were cooled down to room temperature and then placed in the refrigerator for 30 min at -20^{o}C to solidify the sample completely. Finally, the solid sample tubes were cut into three equal sections with the heated knife. The two ends which are top and bottom were extracted from

the container and then tested. Many studies conducted the softening point test to evaluate the storage stability of the binder (Djaffar et al. 2013; de Carcer et al. 2014; Zhu 2016). Recent study showed that the softening point test was unable to give better evaluation of storage stability of the binder on the performance related properties and instead, suggest to use rheological study to analyse the stability of the binder during storage at high temperature (Zani et al. 2017). Thus, the DSR was performed and testing protocol was conducted using similar setting as describe in Section 3.3.6.5.

3.3.3 Penetration Test

Penetration test is a physical test used to measure the consistency or hardness of bitumen at a desired or standard temperature by using Penetrometer (Figure 3.16). This test also can evaluate the thermal susceptibility of bitumen. The procedure of Penetration Test is performed accordance to BS 2000-49: 2015. In the conventional grading system, bitumen was classified regarding to their penetration grade represented by two number namely 60/70. The numbers indicate that a standard needle (under a 50 g load and at 25 C° for 5 seconds) penetrates a distance (in tenth of millimeter) into the asphalt sample between these two numbers. Bitumen with a

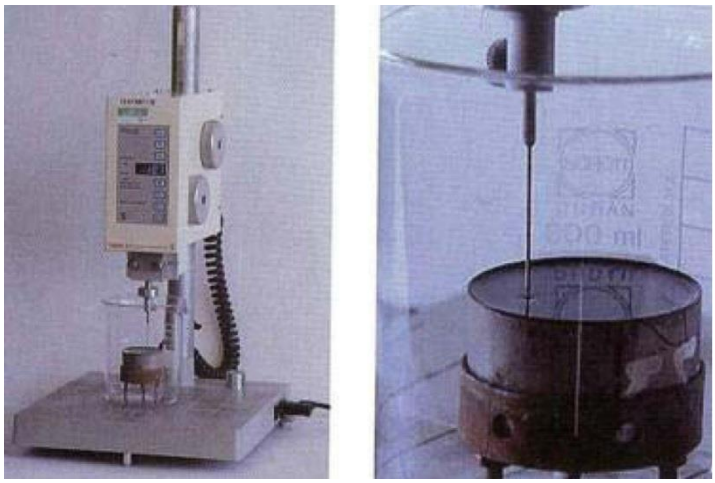


Figure 3.16: The penetration test

lower penetration grade such as 40/50 would be used in a hot region whereas those with higher grade such as 120/150 would be used in a colder region (Mallick & El-Korchi 2013).

3.3.4 Softening Point Test

This is one of the important physical tests for bitumen. The consistency of the bitumen can be determined by measuring the equi-viscous temperature at which the consistency of the bitumen is between solid and liquid behaviour or the temperature of the bitumen starts flowing (Yildirim 2007; Mallick & El-Korchi 2013). Besides that, it has a direct influence on the resistance to permanent deformation of the mixtures (Costa et al. 2013). The test is conducted using the ring and ball apparatus as shown in Figure 3.17 and performed according to BS 2000-58:2015.

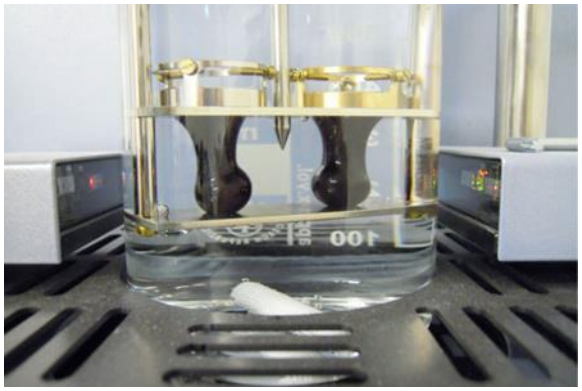


Figure 3.17: The ring-and-ball of softening point test

3.3.5 Viscosity Test

Viscosity is a fundamental characteristic of bitumen as it helps in measuring the resistance of the bitumen to flow. It determines how bitumen will behave at a given stress and shear rate over a range of temperature and can be defined as the ratio between the applied shear stress and the rate of shear strain measured in unit of Pascal seconds (Pa.s). The apparatus used to perform this test is the Brookfield

rotational viscometer and the Thermocel system as shown in Figure 3.18 .The rotational viscometer test method is presently considered to be the most practical means of determining the viscosity of bitumen, since the instrument allows the testing of a wide range of bitumen over a wide range of temperatures, more so than most other viscosity measurement systems (Brown et al. 2009).



Figure 3.18: Rotational viscometer

3.3.6 Dynamic Shear Rheometer

The dynamic shear rheometer (DSR) as shown in Figure 3.19 is a dynamic oscillatory test apparatus that is used to characterise the response behaviour of bitumen (i.e. elastic, viscoelastic and viscous) within the linear viscoelastic (LVE) region when subjected to a load at the wide range of temperatures and loading frequency.



Figure 3.19: Dynamic Shear Rheometer (DSR)

3.3.6.1 Basic Principal

In general, DSR test can be carried out in the two ways which are controlled stress or controlled strain testing modes. For the controlled stress mode, a specified magnitude of shear stress which acts as a fixed torque is applied to the bitumen to oscillate the top plate or spindle and the resultant spindle rotation is measured, from which the magnitude of shear strain is calculated. Controlled strain mode work by applying specified magnitude of spindle rotation and the required torque to cause this movement is measured, from which the magnitude of shear stress is calculated (Petersen et al. 1994). The strain controlled loading normally applied for the dynamic or oscillatory test method while the stress controlled loading normally applied for the transient test methods (e.g. creep, relaxation and constant rate loading) (Subhy 2017a). The principals involved in DSR testing are illustrated in Figure 3.20. The bitumen sample is sandwiched between a spindle and an oscillating plate. When torque moves towards point B, the plate moves back and goes to point C and passing

the point A. Then, from point C it returns back to point A and this sequence of movement comprises one cycle of oscillations (Brown et al. 2009) and continuous cycle which can be continuously repeated during the test. Normally, the DSR test is carried out over a range of frequencies (number of cycles per second) and temperatures.

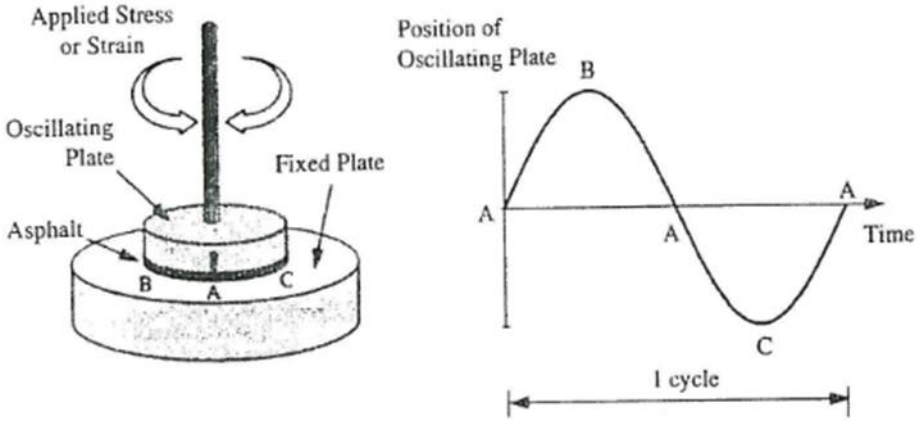


Figure 3.20: Basic of Dynamic Shear Rheometer (DSR) (Brown et al. 2009)

The DSR software used following formulas to calculate stress and strain.

$$\sigma = \frac{F}{A} = \frac{2\,T}{\pi r^3} \qquad \text{Equation 3.3}$$

$$\gamma = \frac{\theta\,\mathrm{r}}{h} \qquad \text{Equation 3.4}$$

Where,

σ = maximum shear stress

F = Force

A = Area

T = torque

r = radius

γ = maximum shear strain

Θ = deflection angle

h = gap between parallel disks

Based on the Equations 3.3 and 3.4, the shear stress and strain parameters are dependent on the radius of the parallel disk and gap between the upper and lower parallel plates. These geometric properties of the oscillating plates are depending on the test temperature and the expected stiffness of the materials tested. There are two main sizes of plate (Figure 3.21) that commonly used which are 8 mm parallel plates with a 2 mm testing gap and 25 mm parallel plates with a 1 mm testing gap. Generally, at low temperature testing and increased in stiffness, smaller radius of plate is used for performing the test and vice versa because of the torque limitation of the DSR (Jiménez del Barco Carrión 2017). The SHRP research team suggest that the following guideline of different plate sizes should be used (SHRP 369):

- Use torsional bar geometry or Bending Beam Rheometer (BBR), for temperature < 0^{o}C when G* > 30 MPa.
- Use 8 mm parallel plates with a 2 mm gap, for temperature range 0^{o}C to 40^{o}C, when 0.1 MPa < G* < 30 MPa.
- Use 25 mm parallel plated with a 1 mm gap, for temperature range 40^{o}C to 80^{o}C when 1.0 kPa < G* < 100kPa
- Use 50 mm parallel plates, for temperature > 80^{o}C. when G* < 1 kPa

Airey stated that it is possible to use the same testing geometry over a wide range of temperature, however, the precision of the results may be limited as a result of compliance errors and the reduction in precision with which the torque can be measured at low-stress levels (Airey 2002; Airey 2002a). He added that over the wide

frequency sweep and for the different bitumen, care should be taken when using these geometries and the rheological testing result of these two plate and gap configurations should be overlap at the transition points (Airey 1997).

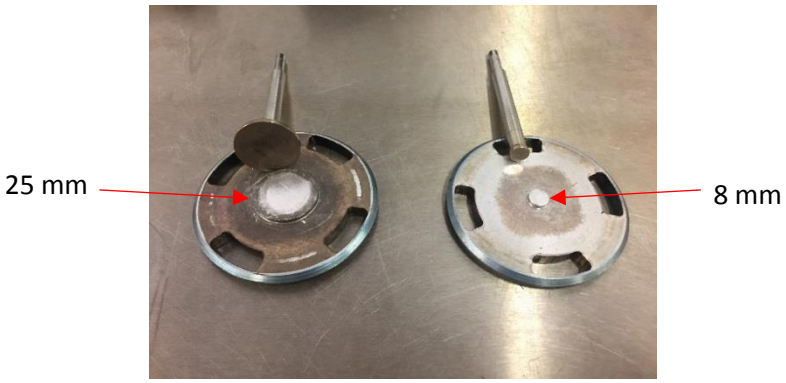


Figure 3.21: Geometries of different DSR spindle

3.3.6.2 Rheological Analysis

The rheological properties of bitumen can be characterised using the Dynamic Mechanical Analysis (DMA) by means of the DSR. The testing methods used for measuring these rheological properties is oscillatory test with the controlled strain condition. This test is performed by applying the sinusoidal strain on the material. The material is subjected to varying sinusoidal strain with a loading frequency (ω) can be expressed as below:

$$\gamma^* = \gamma_0 \sin \omega t \qquad \text{Equation 3.5}$$

Where:

γ^* = dynamic oscillating shear strain

γ_0 = peak shear strain

ω = angular frequency (rad/sec)

t = time (seconds)

The loading frequency (ω) is known as the angular frequency, rotational frequency or radian frequency. It can be defined as:

$$\omega = 2\pi f \qquad \text{Equation 3.6}$$

Where:

f = frequency (Hz) = $1/T$

The response of the applied sinusoidal strain is the sinusoidal stress but will be out lag by an amount of phase angle (δ). Therefore, the response sinusoidal stress is:

$$\sigma^* = \sigma \sin(\omega t - \delta) \qquad \text{Equation 3.7}$$

Where:

σ^* = dynamic oscillating shear stress (Pa)

σ = peak stress (Pa)

δ = phase angle (degree)

The phase angle (δ) is also called as loss angle or phase lag is a function that used for describing the viscoelastic properties of materials. It can be defined as the phase or lag difference between the applied stress and the strain response subjected to a sinusoidal oscillation. The materials are considered purely elastic materials if the phase angle is 0^o whereby the waveform of applied strain is in the same phase with the waveform of response stress. While the materials are purely viscous materials when the waveform of response stress is shifted 90^o from the waveform of applied strain. Meanwhile, the viscoelastic materials produced the phase angle between 0^o to 90^o. Figure 3.22 displayed the schematic stress response to oscillatory strain applied for a purely elastic, a purely viscous and a viscoelastic material.

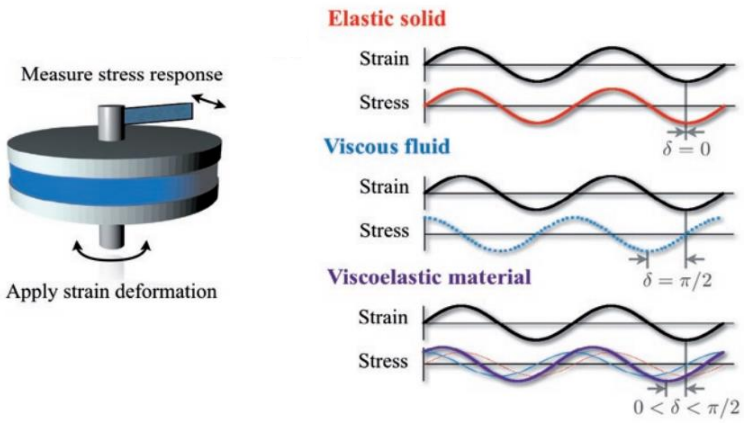


Figure 3.22: Schematic stress response to oscillatory strain deformation for an elastic solid, a viscous liquid and a viscoelastic material

The ratio between the resulting stress and the applied strain is called the complex shear modulus (G*) and defined by:

$$G^* = \frac{\sigma^*}{\gamma^*} = \frac{\sigma_0}{\gamma_0} e^{i\delta} \qquad \text{Equation 3.8}$$

Where:

G^* = complex shear modulus (Pa)

The absolute function or the norm of G^* is the ratio of the peak stress to the peak strain:

$$|G^*| = \frac{\sigma_0}{\gamma_0} \qquad \text{Equation 3.9}$$

Equation above can be written as

$$G^* = \left(\frac{\sigma_0}{\gamma_0}\right) \cos\delta + i \left(\frac{\sigma_0}{\gamma_0}\right) \sin\delta \qquad \text{Equation 3.10}$$

And

$$G^* = G' + iG'' \qquad \text{Equation 3.11}$$

Where:

G' = the storage modulus (Pa)

G'' = the loss modulus (Pa)

From Equation above, the norm of the complex shear modulus can be calculated as the square root of the sum of square of the storage modulus and the loss modulus as follows:

$$|G^*| = \sqrt{(G')^2 + (G'')^2} \qquad \text{Equation 3.12}$$

The complex shear modulus is known as the complex modulus. It can be considered as the stiffness of the material or in details, the total resistance of the binder to deform when repeatedly sheared.

The norm of the complex shear modulus is having the shear storage modulus as the real part or the in-phase component of G^* and the shear loss modulus as the imaginary part or the out-pf phase of G^*. Those function can be expressed as:

$$G' = G^* \cos \delta \qquad \text{Equation 3.13}$$

$$G'' = G^* \sin \delta \qquad \text{Equation 3.14}$$

The shear storage modulus (G') is the elastic component of the complex modulus or the recoverable part and generally known as the storage modulus or elastic modulus. It can be described as the ability of the materials to store energy and released elastically in each oscillation. The shear loss modulus (G'') is referred as the viscous component or the non-recoverable part of the complex modulus. It also known as the loss modulus or the viscous modulus. The loss modulus can be defined as the dissipation of energy or in other words, the energy which is lost for each oscillation in each cycle (Airey 1997).

The ratio of the viscous and elastic component of the complex modulus is the loss tangent or the tangent of the phase angle and be written as:

$$\tan \delta = \frac{G''}{G'} \qquad \text{Equation 3.15}$$

For asphalt binder, the value of G^* and δ are significantly affected by the temperature and frequency of loading. At very low temperature and high loading frequencies, the binder behaves like elastic solids while it will behave like viscous fluids at high temperatures and low frequencies of loading.

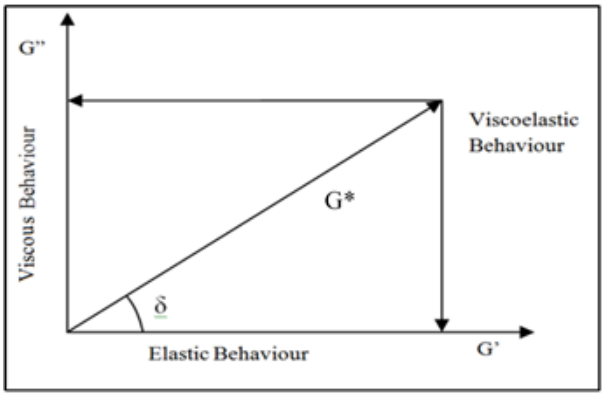


Figure 3.23: The relationship between complex modulus, storage modulus, loss modulus and phase angle

3.3.6.3 Strain Limit

The oscillatory test normally performed within the linear viscoelastic (LVE) region to ensure that the rheological response of the material tested is within the region, on the other hand, independent of the applied strain or load. The viscoelastic response of materials would not be influenced by the magnitude of the applied strain or load, otherwise only influenced by temperature and loading time (Subhy 2017a). Hence, the applied strain must be kept as small as possible for remaining in the LVE region. However, the region will vary with the complex modulus of the binder and thus, at low temperatures, the shear strain should be small while increased at high temperatures. The strain limit of the material could be obtained by conducting the strain (or amplitude) sweep test. During testing, the sample will be subjected to the shear strain that is gradually increased at a fixed frequency and the graph of complex

modulus (G*) versus shear strain is plotted to obtain the LVE region of the material. Regarding to this graph, the strain limit of the material is defined as the point where the response value of G* decreases to 95% of its maximum value as shown in Figure 3.24.

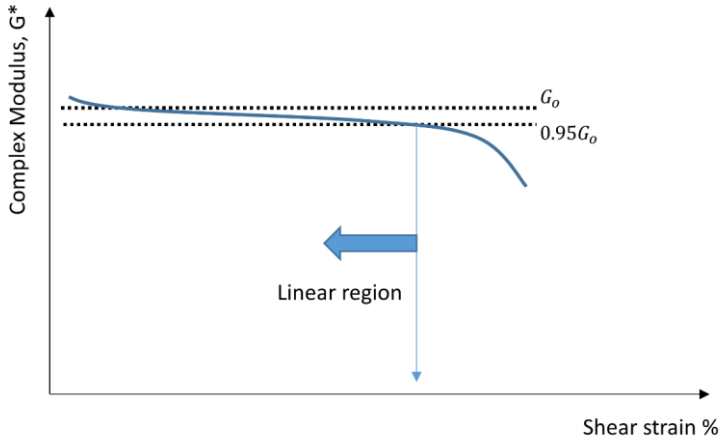


Figure 3.24: Strain sweeps used to determine LVE region

3.3.6.4 DSR Data Representation

There are five common data representations could be used and generated from the DSR result to evaluate the rheological properties of the material namely:

- **Isochronal Plot**

A plot of any bituminous material properties (i.e. complex modulus or phase angle) versus temperature at constant frequency or loading period as displayed in Figure 3.25. This plot can be used to assess the temperature susceptibility of the material used at fixed loading frequency.

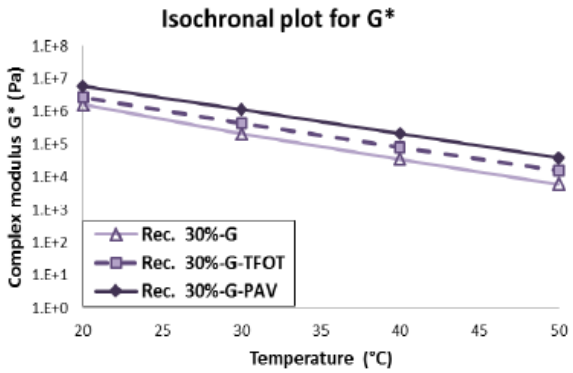


Figure 3.25: Typical isochronal plot for complex modulus of recovered bitumen from granite mastics after different ageing stages (Rami Mohammed 2018)

- **Isothermal Plot**

This plot as shown in Figure 3.26 consists of a curve of any bituminous material properties versus frequency at a constant temperature. This plot can be used to assess the frequency loading susceptibility of the material used at fixed temperature.

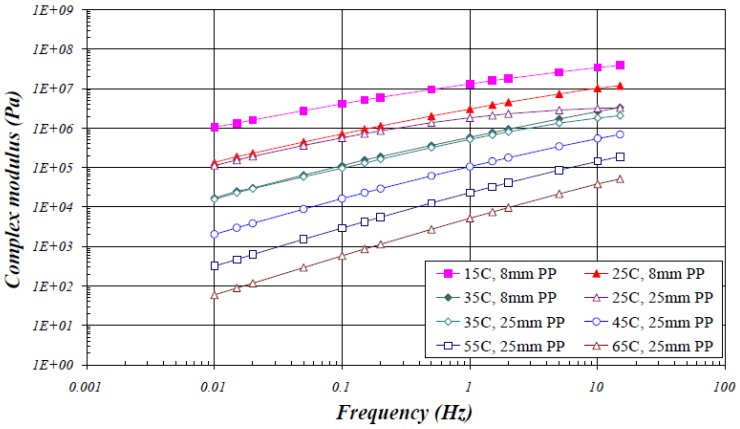


Figure 3.26: Typical isothermal plot of complex modulus for conventional 50 penetration grade bitumen (Airey 2002a)

- **Black Diagram**

A black diagram are curves (Figure 3.27) which display the log complex modulus versus phase angles. In addition, Figure 3.27 displayed the evolution of the G* with δ at different temperatures. Mangiafico (2014) stated that this plot provides a useful

information on the behaviour of the bituminous material at high temperature or low frequency. The behaviour of thermo-rheological simple of the bituminous material can be identified by this diagram in which the binders that is thermorheologically simple displayed a smooth overlapped curve in black diagram as shown in Figure 3.27. However, the higher content of wax, asphaltene structured and polymer could display disjoint curve of black diagram. Moreover, Airey (2002a) stated that the smooth curve of black diagram may not continuously overlap if the measurement of the rheological properties is conducted outside the LVE region or with the selection of inappropriate disk configuration (DSR spindle geometries) for testing.

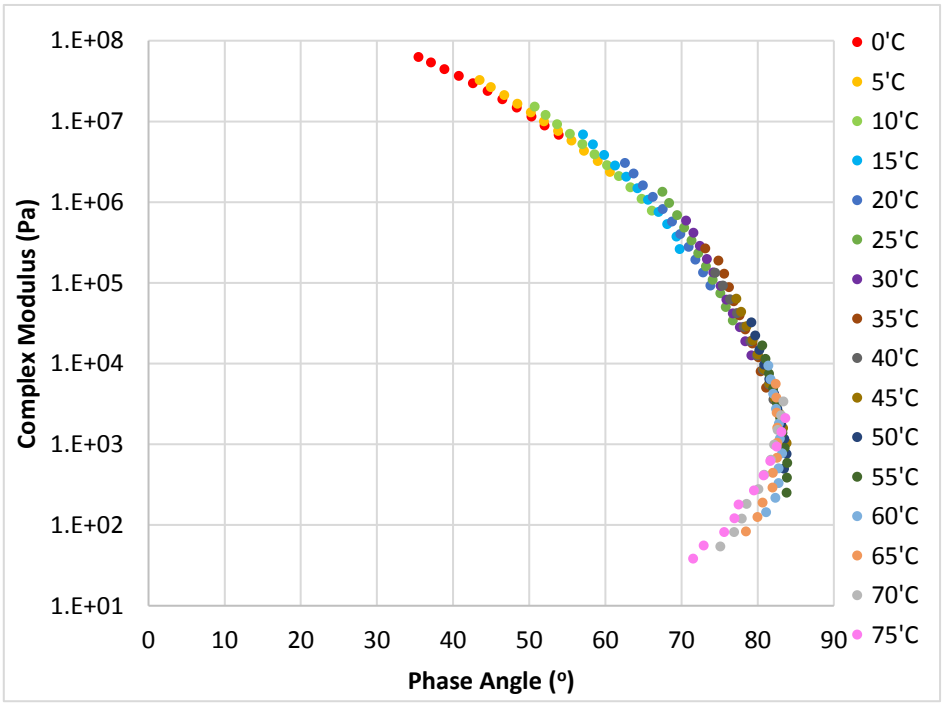


Figure 3.27: Typical black diagram of modified binder with low percentage

- **Cole-cole Diagram**

Cole-cole diagram is a plot of storage modulus versus loss modulus as shown in Figure 3.28. The curve represents the viscoelastic balance of the binder without being

influence by the frequency and the temperature. The behaviour of binders and mixtures at low temperature or high frequency is highlighted by this plot (Mangiafico 2014).

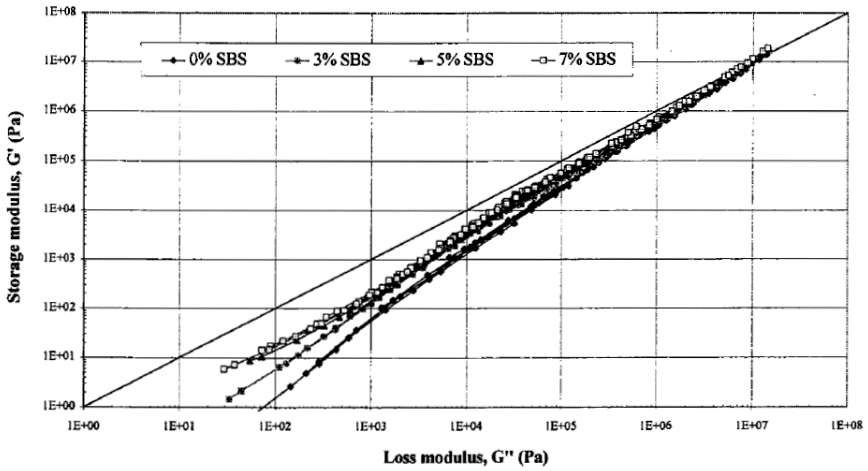


Figure 3.28: Typical cole-cole diagram for SBS PMB (Airey 1997)

- **Master Curve**

The master curve is a plot of complex modulus or phase angle against reduced frequencies on a log-log scale at a particular reference temperature. Thus, the viscoelastic behaviour of the binder could be assessed at an expanded range of loading frequencies. The smooth and continuous plot of the master curve is obtained from the isothermal plots that are manually shifted by using a different shift factor $a(T)$ for each temperature to a reference temperature. The Williams, Landel and Ferry (WLF) equation has been used to obtain the shift factor by using the following equation:

$$Log\ a_t = \frac{C_1(T - T_{ref})}{C_2\,(T + T_{ref})} \qquad \text{Equation 3.16}$$

Where:

a_t = shift factor

C_1 and C_2 = constant

T = temperature

T_{ref} = reference temperature

For constructing a smooth master curve, C_1 and C_2 were obtained using Solver in Microsoft Excel for the optimal calculation. Then, a reference temperature is selected and the isotherm plots are shifted along the frequency axis with a distance of log $a(T)$ as presented in Figure 3.29.

Nevertheless, the discontinuous shape of master curve could occur especially for waxy bitumen, polymer modified binder and highly gel-type bitumen due to a structural change in the binder with temperature (Airey 1997; Di Benedetto et al. 2011; Asgharzadeh & Tabatabaee 2013).

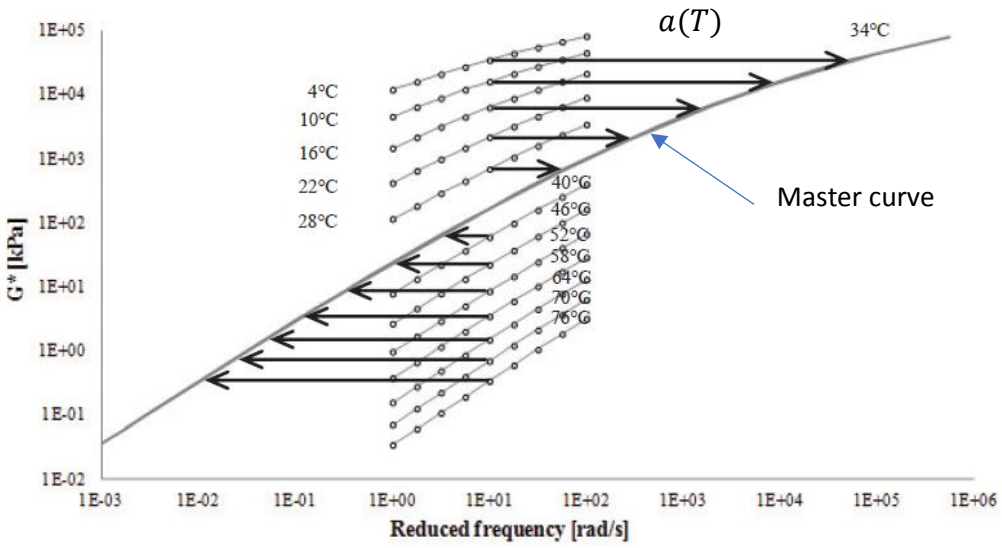


Figure 3.29: Typical isothermal plot and the master curve construction with shift factor at reference temperature of 34oC (Santagata et al. 2012)

3.3.6.5 Testing Protocol

The unaged and aged binders were subjected to the strain sweep test and frequency sweep test. These tests were conducted by means of CVO Dynamic Shear Rheometer (DSR). The strain sweep test usually undertaken before running the frequency sweep test for obtaining the LVE strain limit of the binders. The strain sweep test was

conducted at temperature of 10^{o}C for geometry 8mm parallel plate and 40^{o}C for geometry 25mm parallel plate. Once the LVE strain limit of the binders was defined, the frequency sweep test was carried out within that limit. The test parameter used for frequency sweep test were as follows:

- Mode of loading: strain-controlled
- Temperature: 0^{o}C – 75^{o}C at 5^{o}C intervals
- Frequencies: 0.1 – 10 Hz
- Spindle geometries: 8 mm diameter plate and 2 mm gap (0^{o}C to 35^{o}C) and 25 mm diameter plate and 1 mm gap (25^{o}C to 75^{o}C)
- Strain amplitude: within the LVE region (1% strain for both spindle geometries)

3.3.7 Rolling Thin Film Oven Test (RTFOT)

In this study, the RTFOT was used to simulate the hardening of bituminous material for short-term ageing that occurs during the construction process. This RTFOT was carried out in accordance to BS EN 12607-1:2014. The test involved of eight glass bottles (as shown in Figure 3.30) whereby each bottle containing 35 g of bitumen. Then, all eight glass bottles were placed into a vertically rotating shelf in the RTFO oven (Figure 3.31) at a temperature of 163^{o}C for 75 minutes. The shelf was rotated continuously at the rate of 15 rpm to avoid a formation of surface skin for inhibiting the bitumen from ageing and the air flow into the bottles was maintained at 4000 mL/min for exposing the bitumen to heat and air (Airey 1997; Airey 2003).



Figure 3.31: RTFO glass bottles



Figure 3.30: RTFOT oven

3.3.8 Pressure Ageing Vessel (PAV) Test

The long-term ageing of bitumen that occurs during in-service ageing in the field can be simulated by using PAV test. This test was used to age RTFOT residue because the bitumen in the pavement has experienced short-term ageing during construction. The PAV procedure were done according to BS EN 14769:2012. The amount of 50 g of bitumen was poured in a 140 mm diameter pan and placed into PAV machine by using a pan holder as shown in Figure 3.32. In the machine, the bitumen was aged for 20 hours at temperature of 100^{o}C in a heated vessel pressurised with air up to 2.10 MPa.



Figure 3.32: PAV Machine

3.4 Performance Characteristics Test for Binders

Fatigue failure usually occur in the intermediate temperature ranging from 10^{o}C to 30^{o}C when the asphalt pavement subjected to the repeated loading (Al-Khateeb & Shenoy 2004). With a weak the pavement structural due to the repeated application of stresses (traffic loading), crack initiation is taking place at the bottom of the pavement layers where tensile strain is highest and then propagate upward to the surface (Mashaan et al. 2014). However, variation of temperature and construction practice could contribute to this deterioration.

Rutting or permanent deformation in asphalt mixture normally occurs at high temperatures (>40^{o}C) when the binder is soft and viscous behaviour (Al-Khateeb & Basheer 2009). Rut appears along the wheel path due to the plastic deformation of the asphalt pavement subjected to the increasing number of load repetitions (Jiménez del Barco Carrión 2017).

Durability of asphalt mixture is defined as the ability of the compacted material in asphalt mixture to sustain the integrity of its structure and properties when exposed to the traffic loading and environmental condition throughout its expected service life (Nicholls et al. 2008; Bonaquist 2013). Thus, road pavement that is good in durability performance is demanded in order to minimize maintenance cost. However, ageing and water damage could weaken the asphalt mixture gradually.

Therefore, the evaluation of these pavement deteriorations is vital for ensuring that the quality of the road pavement is improved. The performance of binder has been carried out in term of fatigue, rutting and durability of the binder at dry and wet condition. Two methods were used to characterise the fatigue resistance which are Linear Amplitude Sweep (LAS) test and Double-Edged Notched Tension (DENT) test

while the Multi Stress Creep Recovery (MSCR) is used to evaluate the resistance to rutting. Then, the durability of the bituminous paving mixtures in term of fracture property of mineral aggregates bound with a binder were examined by means of the Peel test.

3.4.1 Linear Amplitude Sweep (LAS) Test

For many decades, the SHRP fatigue parameter namely G*sin δ has been used as an indicator to evaluate the fatigue resistance of asphalt binder in the linear viscoelastic (LVE) properties and primarily developed for unmodified binders (Hintz et al. 2011; Zhou et al. 2012). It is a stiffness-based parameter which involved the relationship between the loss modulus and the energy dissipated per cycle (Bahia et al. 2001) whereby large values of G* sin δ is undesirable because it indicates that the dissipated energy per loading cycle is increased and it will dissipate by producing crack in the material (Johnson 2010). It measured the distresses at very small shear strain (LVE region) for few cycles of loading which means that the properties of the asphalt binder is not affected by the strain when the measurement is obtained (Zhou et al. 2012) or in other word, the damage still not occurred. Unfortunately, the G*sin δ is not relevant to be used for indicating resistance to fatigue damage as fatigue phenomenon becomes more complicated to predict in the real situation on the road such as increase in traffic volume required more cycles of loading to be tested and therefore actual fatigue damage resistance could not be characterised and the poor correlation between fatigue life of asphalt mixture and this parameter at intermediate temperature (Bahia et al. 2001). Moreover, the modified bitumen exhibits better resistance to fatigue cracking and show a nonlinear response to loading, thus this parameter is not applicable.

To overcome these deficiencies, Bahia et al. (2001) proposed time sweep test in NCHRP report 459 to characterise the fatigue behaviour of binders. The time sweep tests is based on definition of fatigue damage namely the material degradation under repeated loading and it performed using DSR by applying repeated cyclic loading of strain or stress at fixed loading frequency until failure (Hintz et al. 2011). The good correlation has been made between time sweep test and asphalt mixture fatigue test. Unfortunately, the test procedures was impractical due to long testing time and also unrepeatable (Johnson 2010). Thus, in 2010, Johnson developed new test to measure fatigue performance known as Linear Amplitude Sweep (LAS) that involved an accelerated fatigue procedure and less time to perform. A year later, Hintz et al. (2011) have modified the LAS test procedure and then, the correlation between the LAS result with asphalt mixture and field performance have been obtained (Hintz et al. 2011; Zhou et al. 2012; Saboo & Kumar 2016). Besides that, the procedure of LAS test has received approval from the FHWA Binder ETG in 2011 and designated a provisional AASHTO standard procedure under AASHTO TP101-12 in 2012 (current version is TP101-14).

3.4.1.1 Test Procedure

According to AASHTO TP 101-14, the purpose of LAS is '*to evaluate the ability of an asphalt binder to resists damage by employing cyclic loading at increasing amplitudes in order to accelerate damage'*. LAS is performed on binder that has been aged in the rolling thin film oven (RTFO) and tested using the Dynamic Shear Rheometer (DSR) and 8 mm parallel plate geometry with a 2 mm gap setting. It consists of two types of testing which are frequency and amplitude sweep. The first test, the frequency sweep, is performed by applying oscillatory shear load of 0.1 percent strain over a

range of loading frequencies from 0.1 to 30 Hz to obtain the undamaged rheological properties of the binders which is alpha (α) parameter. This α parameter can be defined as the material constant related to the rate at which damage progress (Zhou et al. 2012) and important for determining the parameter B in the fatigue law. The second test which is amplitude sweep test is executed in strain control mode at a frequency of 10 Hz with the strain amplitude is increased linearly from 0.1 to 30 percent. The purpose of the strain sweep is to measure the damage characteristics of the material. From amplitude sweep test, the parameter α of the fatigue law can be obtained through viscoelastic continuum damage (VECD) analysis and then, the fatigue resistance of the asphalt binder can be calculated. The parameter used for performing LAS test by means of DSR are as follow:

The frequency sweep test:

- Temperatures: 15, 20 and 25^{o}C.
- Strain level: 0.1% over a range of frequencies
- Frequencies: 0.2, 0.4, 0.6, 0.8, 1.0, 2.0, 4.0, 6.0, 8.0, 10, 20 and 30 Hz
- Spindle geometries: 8mm diameter parallel plates with 2 mm gap.
- Sample tested: RTFOT+PAV ageing (to simulate the ageing during the service life of pavement).

The amplitude sweep test:

- Temperatures: 15, 20 and 25^{o}C.
- Mode of loading: strain-controlled from zero to 30%
- Frequency: 10 Hz
- Spindle geometries: 8mm diameter parallel plates with 2 mm gap.

- Sample tested: RTFOT and then PAV ageing (to simulate the ageing during the service life of pavement).

3.4.1.2 Analysis Procedure

LAS test result is analysed by using VECD which based on Schapery's theory of work potential to model damage growth (Zhou et al. 2012). Based on this theory, the damage rate (the available force for damage growth) is defined as the change of energy potential of material (W) with respect to the change of damage (D) in the material and Equation 3.17 showed this relationship (Zhou et al. 2012):

$$\frac{dD}{dt} = \left(-\frac{\partial W}{\partial D}\right)^{\alpha} \qquad \text{Equation 3.17}$$

Where:

$t =$ time

$W =$ work performed

$D =$ damage intensity

$\alpha =$ material constant related to the rate at which damage progresses

The LAS test is carried out in two types of testing on the same sample of material. The first test, frequency sweep test, is conducted at low shear strain level which is 0.1% to avoid damaging the sample with frequency range of 0.1 to 30 Hz. During this test, the value of complex modulus ($G*$), phase angle (δ) and storage modulus (G') obtained were used to determine the damage exponent (α) parameter. The α is calculated by using the slope of a log-log plot of relaxation modulus versus time (Hintz et al. 2011). This parameter will be used later on in the analysis of amplitude sweep data.

In order to determine α, the method of converting the frequency sweep data to relaxation modulus has been developed by Johnson (2010) based on the inter-

conversions method (Schapery & Park 1999). Later, modification in calculating α has been made by Hintz et al. (Hintz et al. 2011) and they found that the slope of a log-log plot of G' versus frequency (ω) can be used to calculate α.

Parameter α is the reciprocal of the straight line slope (m) of $log\ G'\ (\omega)$ versus $log\ \omega$ curve (Saboo & Kumar 2016). The example of the resultant plot is shown in Figure 3.33 and an equation can be formed as Equation 3.18. From the equation, the value of m is recorded.

$$\log G'(\omega) = m\ (\log \omega) + b \qquad \text{Equation 3.18}$$

Then, the α value is calculated by using the following transformation:

$$\alpha = \frac{1}{m} \qquad \text{Equation 3.19}$$

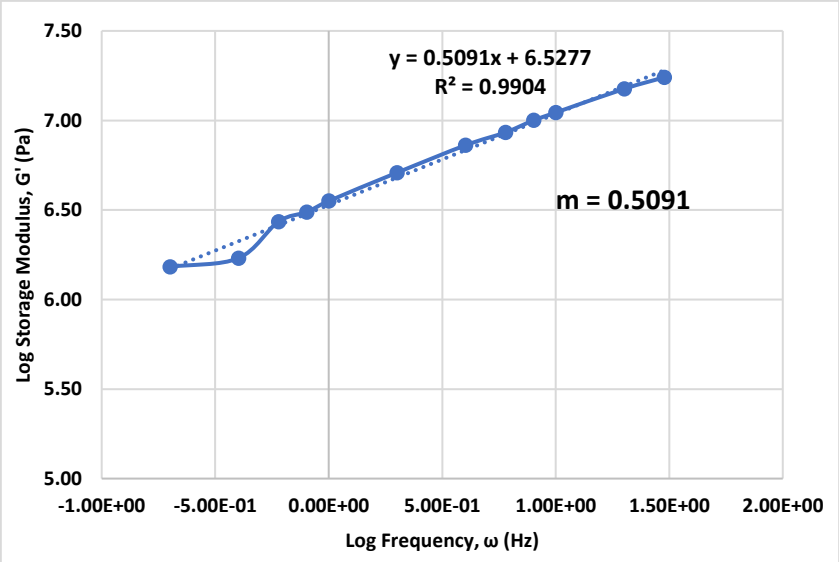


Figure 3.33: Calculation of α

After obtaining the α value, the second test namely amplitude sweep test is performed directly after the frequency sweep finished. The strain level starts at 0.1% and ends at 30% with an increase of 1% strain over the constant frequency of 10 Hz. The sample is subjected to 100 cycles for each strain level. The output of amplitude

sweep test which are $G*$, δ, shear stress (τ), strain (γ) and time (t) were recorded to perform the analysis.

The damage accumulation in the specimen is calculated for each data point by using the Equation 3.20.

$$D(t) \cong \sum_{i=1}^{N} [\pi \gamma_0^2 \, (C_{i-1} - C_i)]^{\frac{\alpha}{1+\alpha}} (t_i - t_{i-1})^{\frac{1}{1+\alpha}} \qquad \text{Equation 3.20}$$

Where:

$C(t) = \frac{|G^*|(t)}{|G^*|_{initial}}$ = $|G*|$ at time, t divided by the initial undamaged value of $|G*|$

$\gamma_0 =$ applied strain for a given data point (%)

$|G*| =$ complex shear modulus (MPa)

t = time (s)

The initial undamaged value of $|G*|$ is the second data point of $G*$. Summation of damage accumulation begins with the first data point until the final data point which is when 30 percent of strain is applied. The incremental value of damage accumulation at each subsequent point is added to the value of damage accumulation from the previous point.

Then, the calculated material integrity, $C(t)$ and $D(t)$ are used to fit a relation of the Equation 3.21 below with assumption of C at $D(0)$ is equal to 1 and $D(0)$ equal to 0.

$$C_t = C_0 - C_1(D)^{C_2} \qquad \text{Equation 3.21}$$

$C1$ and $C2$ can be obtained using curve fitting adapted from Hintz et al. (2011) as shown below:

$$\log(C_0 - C(t)) = \log C_1 + C_2 \log(D(t)) \qquad \text{Equation 3.22}$$

Graph of $\log(C_0 - C(t))$ versus $\log D$ is plotted as depicted in Figure 3.34 and a linear equation is produced. Hence, value of C_1 and C_2 can be obtained by linearization of this graph (Figure 3.34) whereas C_1 is the anti-log of the intercept and C_2 is the slope of this line. In Hintz et al. (2011) study, in order to determine value of C_1 and C_2, they recommended to ignore data at damage levels below 100 because the data below 100 showed nonlinear behaviour. However, in this study, by referring to latest AASHTO standard, the damage value that is less than 10 is ignored for calculating C_1 and C_2.

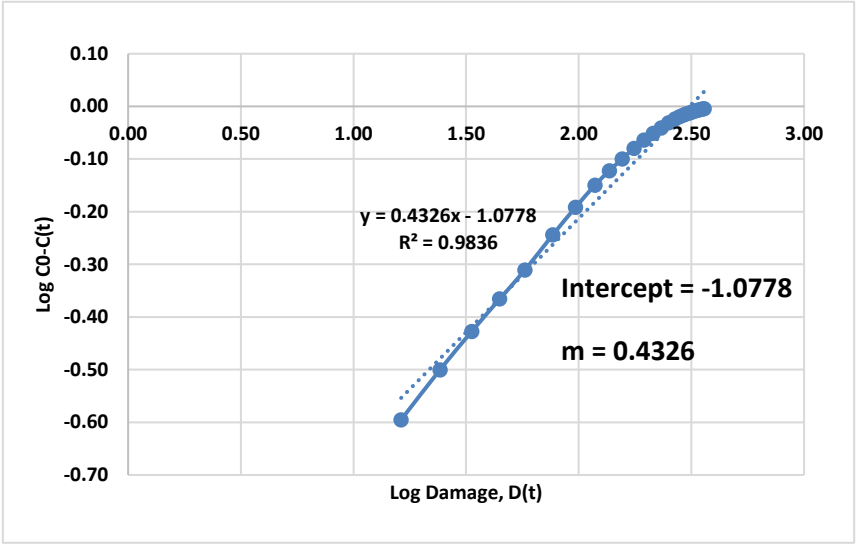


Figure 3.34: Calculation of C_1 and C_2

The value of $D(t)$ at failure (Df) is calculated as follows:

$$D_f = \left(\frac{C_0 - C \text{ at } Peak\ Stress}{C_1}\right)^{\frac{1}{C_2}} \qquad \text{Equation 3.23}$$

The model parameter for the binder fatigue performance namely A and B are evaluated using the following equations:

$$A = \frac{f(D_f)^k}{k\,(\pi C_1 C_2)^{\alpha}} \qquad \text{Equation 3.24}$$

$$B = 2\alpha \qquad \text{Equation 3.25}$$

Where:

f = loading frequency (10Hz)

$k = 1 + (1 - C_2)\alpha$

Finally, the relationship between applied load and binder fatigue life is derived and calculated with

$$N_f = A(\gamma_{\max})^{-B} \qquad \text{Equation 3.26}$$

This model allows for consideration of pavement structure (applied load amplitude) and traffic (number of cycled to failure) (Hintz 2012) and schematic relationship of Equation 3.26 is shown in Figure 3.35.

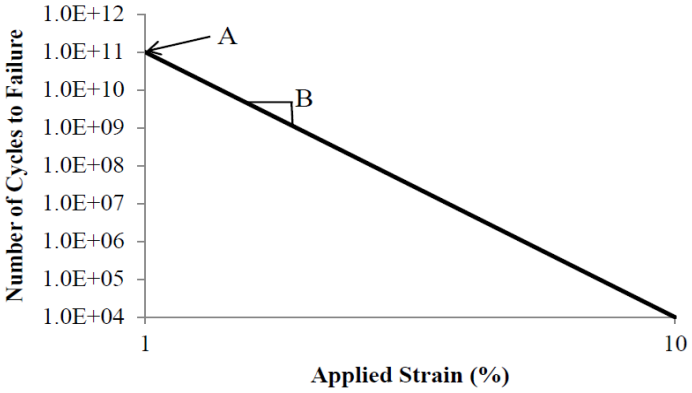


Figure 3.35: Fatigue Law (Hintz 2012)

3.4.2 Double Edge Notched Test (DENT)

A study has been conducted by Andriescu et al. (2004) in Queen's University on characterising the fracture properties of binder by means of the double-edged notched tension (DENT) test based on the concept of essential work of fracture (EWF) of the materials in the ductile state. Many studies reported that this test able to evaluate cracking potential of the binder (Hesp et al. 2014; Shamborovskyy 2016; Singh & Girimath 2016; Subhy et al. 2017; Campbell et al. 2018).

The essential work of fracture (EWF) method and later the crack tip opening displacement ($CTOD$) are used for the fracture toughness assessment of the binder. The EWF method consists of an energy analysis that occurred in ductile failure during the yielding, necking and tearing processes in which this failure processes are closely imitated the way on how binders fail inside an asphalt mixture (Hesp et al. 2014). Wu and Mai (1996) assumed that when a notched ductile specimen is being loaded, the total energy consumed during the fracture can be partitioned into two parts which are the essential work of fracture (W_E) that occurred in the inner fracture process zone and the non-essential work of fracture or plastic work (W_P) that performed in the outer plastic zone as shown in Figure 3.36. The essential work (W_E) is the energy needed to create new fracture surface for progressing the crack and it represents the work done for pulling the thin filament of binder apart in between of two aggregate particles (Subramani 2009). The energy dissipated in the ductility, plasticity and tearing is associated with the plastic work (W_P) whereby plastic deformation takes place. The essential work of fracture is surface related and thus proportional to ligament section area ($L\ x\ B$) while the plastic work is volume related and thus proportional to the volume of the deformed region ($L^2\ x\ B$).

The total work of fracture (W_T) is determined from area under the force displacement curve of these two parts and can be expressed in Equation 3.27:

$$W_T = W_E + W_P \qquad \text{Equation 3.27}$$

The above equation can be written in specific term of energy per unit surface area as follow:

$$W_T = w_e\, L\, B + \beta\, w_p\, L^2\, B \qquad \text{Equation 3.28}$$

By dividing Equation 3.28 with ligament sectional area ($L\ x\ B$), it yields:

$$w_t = \frac{W_T}{L\ x\ B} = w_e\ + \beta\ w_p\ L \qquad \text{Equation 3.29}$$

Where:

w_t = specific total work of fracture (J/m^2)

w_e = specific essential work of fracture (J/m^2)

w_p = specific plastic work of fracture (J/m^2)

B = sample thickness (m)

L = ligament length (m)

w_p= constant that depends on the shape of plastic zone around the failure plane

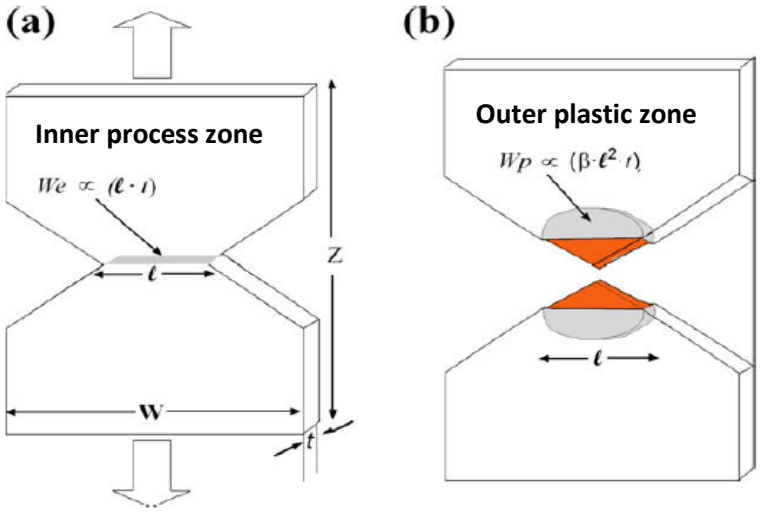


Figure 3.36: Typical dimensions, where the a) the inner process zone (ligament section), b) outer plastic zone are schematised (Martinez et al. 2009)

Based on the Equation 3.29, the specific total work of fracture (w_t) versus the ligament length (L) can be plotted and by performing linear regression of these plot, the intercept of the plot represent w_e (a measure of strength and toughness) and the slope of the line provides (βw_p) can be obtained as shown in Figure 3.37.

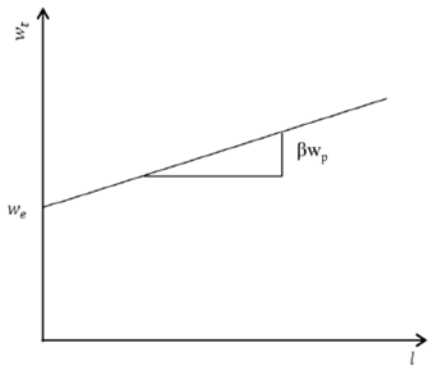


Figure 3.37: The relationship between the specific total work of fracture and ligament length (L) (Subhy 2017a)

Once the w_e is obtained, the critical top opening displacement ($CTOD$) can be determined by using the net section stress of the smallest ligament length tested (σ_n) as follows:

$$\delta_t = \frac{w_e}{\sigma_n} \qquad \text{Equation 3.30}$$

Where:

δ_t = the critical crack tip opening displacement (m)

σ_n = the net section stress (Nm^2) (used here as a replacement for tensile yield stress)

This parameter used the value net section stress of smallest ligament length because it is believed that at the smallest ligament, the yielding of the sample is sufficient to provide a reasonable approximation for the tensile yield stress (Andriescu et al. 2006; Subhy et al. 2017). The $CTOD$ value provide a measure of the strain tolerance (measure how far binder can stretch in a confined space) of the binder in the presence of a crack at the ductile state under severe tensile stress (Andriescu et al. 2006; Andriescu et al. 2009; Subramani 2009; Subhy et al. 2017). Higher value of $CTOD$ is desirable as it corresponds to a good condition of the roads (the pavement can bend more before the crack begins to propagate) and thus provide a longer fatigue life.

Besides that, in order to access the toughness of the binder by using the EWF method, the following conditions should be met (Andriescu et al. 2004; Martinez et al. 2009; Bárány et al. 2010; Subhy et al. 2017):

1) Prior to crack propagation, the ligament must fully yielded.
2) Load-displacement (L-d) diagrams should be self-similar among all different ligament lengths tested, verifying a common geometry of fracture.
3) The samples must be in the plane stress conditions

The test was conducted as following with two replications for each sample:

- Specification: LS-299
- Temperatures: 10^{o}C and 20^{o}C
- Displacement rate: 50 ± 2.5 mm/min
- Sample tested: RTFOT and then PAV ageing (to simulate the ageing during the service life of pavement).

The similar test was performed for different ligament lengths namely 5, 10 and 15 mm as shown in Figure 3.38. The typical raw data from DENT test is depicted in Figure 3.39 in which the area under the force-displacement curve is represent the total work of fracture (W_T).

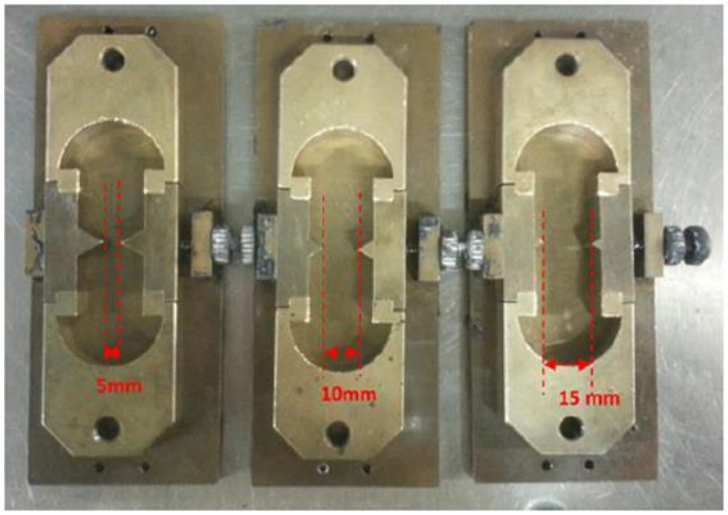


Figure 3.38: DENT test moulds (Subhy 2017a)

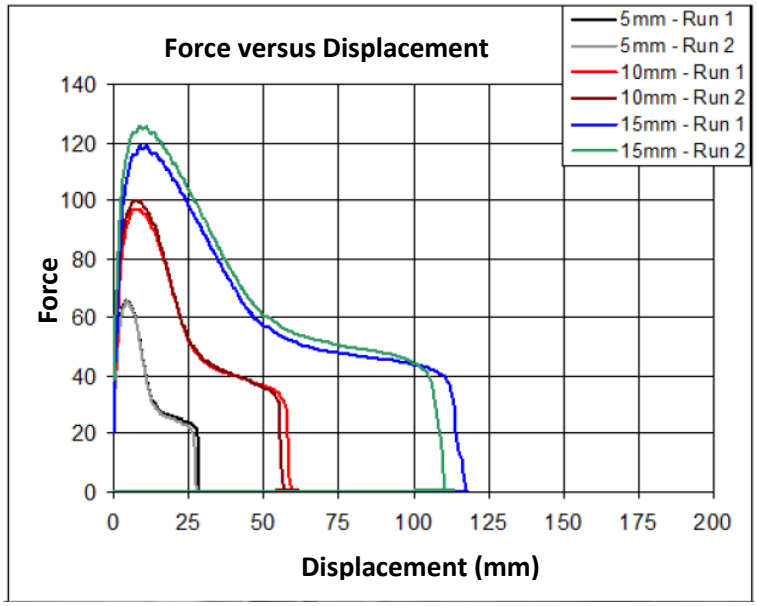


Figure 3.39:Typical raw data of DENT test (Shamborovskyy 2016)

3.4.3 Multiple Stress and Creep Recovery (MSCR) Test

The prediction of pavement rutting can be measured by using the original Superpave binder specification namely the parameter $G^*/\sin\delta$ at 10 rad/s and primary designated for unmodified binder. This parameter is obtained based on stiffness and calculated in the linear region. However, many studies reported that this parameter had poor correlation with field rutting measurements because the rutting occurs in

the nonlinear region in the fields especially for modified binders (Delgadillo et al. 2006b; D'Angelo 2009a; Hossain et al. 2015b). Besides that, the unmodified binder has linear flow and is stress independence which is opposite behaviour with the polymer modified binders in which the chains of polymer tend to rearrange as the stress level increased (Hossain et al. 2015a). Therefore, in 2007, D'Angelo et al. developed a new test to evaluate rutting characteristics of binder known a Multiple Stress Creep and Recovery (MSCR) test.

The MSCR method incorporates the creep and recovery test concept for evaluating the potential of the binder to experience permanents deformation under repeated loading (Zhou et al. 2014). Generally, creep can be defined as deformation under sustained loading whereby for binder, it refers to the changes of material characteristics and properties when subjected to a constant load for a given period of time while recovery is the relaxation after the load applied has been moved completely for a given of time. The susceptibility of the asphalt mixture to rutting could be evaluated using MSCR test by assessing the ability of the binder to recover deformation. Moreover, Kataware and Singh (2017) suggest to perform MSCR test at higher stress level which represents the excessive loading conditions for evaluating the rutting potential and thus, a better characterisation of binder.

3.4.3.1 Testing Protocol

The MSCR test is based on the binder creep and recovery characterisation to evaluate the rutting resistance of the binder. It conducted by using DSR for the parallel plate geometry of 25 mm with a 1 mm gap setting at high temperature and performed according to BS EN 16659-2015 or AASHTO TP 70-13. The sample is subjected to the several stress levels and for each stress level, the sample is loaded to deform for 1

second (creep) and then allowed to recover for 9 seconds (recovery) and these 10 creep-recovery cycles is repeated for 10 times. The parameters used for conducting this test are as follows:

- Temperatures: 40, 60 and 70°C.
- Stress levels: 0.1, 0.4, 1.6, 3.2, 6.4, 12.8 and 25.6 kPa.
- Spindle geometries: 25 mm diameter parallel plates with 1 mm gap.
- Sample tested: RTFOT ageing (to simulate the ageing during mixing and lay-down).

The performance parameter obtained to evaluate the rutting resistance of binders from this test are the non-recoverable creep compliance (J_{nr}) and the percentage of recovered strain or the percent recovery ($R\%$). According to Hossain et al. (2015a) The J_{nr} is defined as a measure of the amount of residual strain left in sample after repeated creep and recovery while the $R\%$ measure of how much the binder able to return to its original shape after being repeatedly stretched and relaxed. Thus, binder with a low J_{nr} and a high $R\%$ is desirable for acceptable rutting performance. Besides that, the J_{nr} can be used to characterise the susceptibility of the binder to the change in stress level especially for polymer modified binder which make it more recommended than the SHRP parameter $G^*/\sin\delta$ for evaluating the rutting behaviour of the binder in asphalt mixture (D'Angelo & Dongré 2009b). From a typical one cycle of creep and recovery as shown in Figure 3.40, the value of these parameter for example at stress applied of 100 Pa and 3200 Pa is calculated as:

$$J_{nr\ at\ 100Pa}\left({}^{1}/_{KPa}\right) = \frac{1}{10}\left\{\sum_{i=1}^{10}\frac{\gamma_{(nr)i}}{0.1}\right\} \qquad \text{Equation 3.31}$$

$$J_{nr\ at\ 3200Pa}\left({}^{1}/_{KPa}\right) = \frac{1}{10}\left\{\sum_{i=1}^{10}\frac{\gamma_{(nr)i}}{3.2}\right\} \qquad \text{Equation 3.32}$$

$$R\%\ at\ 100\ Pa\ or\ 3200\ Pa = \frac{1}{10}\left\{\sum_{i=1}^{10}\frac{\gamma_{(r)i}}{\gamma_{(t)i}}\right\}\ x\ 100 \qquad \text{Equation 3.33}$$

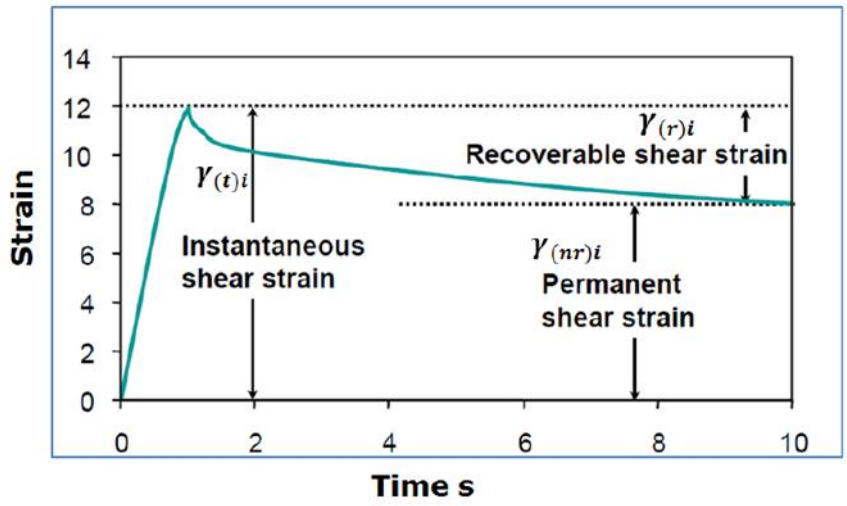


Figure 3.40: Typical schematic of one cycle creep and recovery in MSCR test (Subhy 2017a)

Figure 3.41 a) and Figure 3.41 b) showed an example of the schematic representation of the MSCR test loading and unloading sequence for different stress level and the typical result from the test, respectively.

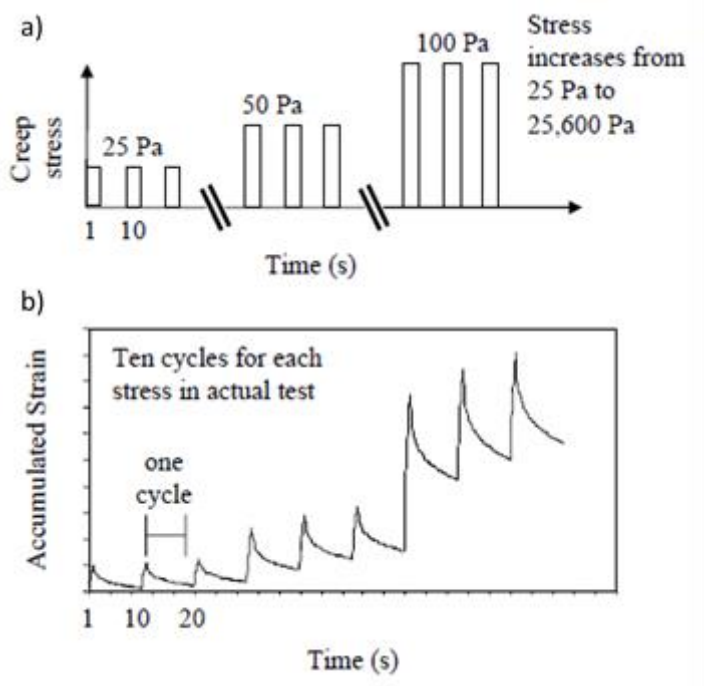


Figure 3.41: The schematic representation of a) MSCR test loading and unloading sequence, b) MSCR response curve (Wasage et al. 2011)

3.4.4 Durability Property – Fracture resistance

The adhesive/cohesive fracture property of mineral aggregates bound with a binder is known as one of the main properties of asphalt mixture on the road pavement. Strong and durable bituminous paving mixtures is needed under all conditions to ensure that the road pavement could be long-lasting. However, on the field, the road pavement is exposed to the age hardening and moisture damage in which these are two primary factors that could affect the durability of the asphalt mixture (Airey et al. 2005). Ageing could make the stiffness modulus of binder increase and the water damage could lead to the cohesion or adhesion loss of the aggregate-bitumen system. Thus, the study on the fracture properties of the aggregate-bitumen interface is vital. Blackman et al. (2013), Zhang et al. (2015b), Zhang et al. (2016a) and Zhang et al. (2016b) used the tensile force and fracture energy of the peel sample to evaluate the degree of cohesion/adhesion of the sample to assess the durability of asphalt pavement materials. Previous studies have shown that the Peel test could characterise the fracture resistance of the aggregate-bitumen interface successfully (Blackman et al. 2013; Zhang et al. 2015b; Zhang et al. 2016a; Zhang et al. 2016b).

3.4.4.1 Testing Protocol

In this study, the composite substrate peel test (CSPT) was used for testing as the CSPT is considered to be reliable for evaluating the aggregate-bitumen bond because the coarse aggregate used in CSPT is similar with the actual asphalt mixture in the field. Besides that, Zhang et al. (2016a) found the strong correlation between the standard peel test and the CSPT for the evaluation of moisture damage. The substrate

preparation for CSPT was done as describe in Zhang et al. (2016a). Then, the sample preparation for the peel test consists of the following steps:

- The substrate is wiped using towel to remove the dust.
- Then, the substrate is placed in the oven for 30 minutes at temperature of 50^{o}C to remove the surface moisture. While the aluminium peel arm is heated at 150^{o}C for 10 minutes. The binders used for testing is heated at 150-190^{o}C.
- At one edge of the substrate, the release film (PTFE) with dimensions of 30 mm x 20 mm x 12.7 µm is placed.
- Five wire spacers with a diameter of 20 mm are placed equally on the aggregate for controlling the uniform thickness of the bitumen layer.
- Along the surface of the substrate, the liquid bitumen is applied uniformly and once finished, the heated aluminium peel arm is placed on the top of the bitumen layer. To ensure that the thickness of the bitumen layer is uniform, a gentle pressure is applied and the pressure should be uniformly distributed over the surface.
- Lastly, all samples were stored at a temperature of 20^{o}C for overnight prior to testing.
- Before running the test, the extra bitumen and aluminium peel arm are trimmed with the sharp knife and the prepared sample is shown in Figure 3.42.

The peel test was performed at room temperature of 20^{o}C with the peel rate of 10 mm/min by using the universal testing machine (UTM) and the test configuration is depicted in Figure 3.43 this machine is capable to supply a constant rate of grip separation and measure the tensile force and displacement at the same time during the test. Figure 3.44 shows an example of the result during the peel test.



Figure 3.42: The peel test sample

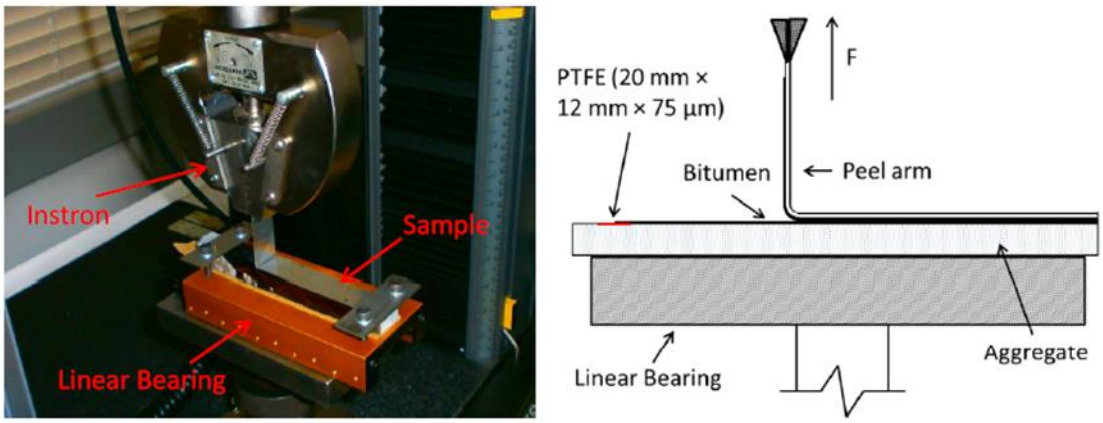


Figure 3.43: Details of peel test configuration (Zhang 2015)

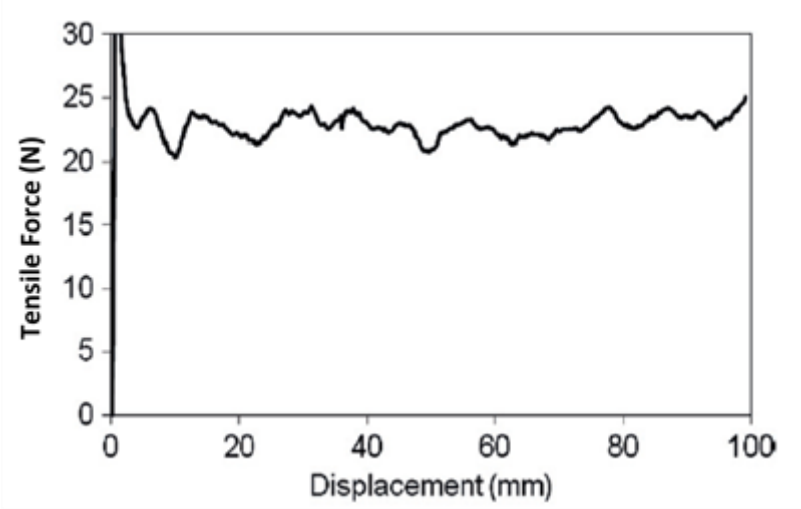


Figure 3.44: Typical raw data from Peel test

From the result obtained in the peel test, the fracture energy of aggregate-bitumen bonds can be calculated by using software known as 'ICPeel' as shown in Figure 3.45 that was developed at Imperial College London based on the fracture theories. The details of fracture theories can be found in Zhang (2015).

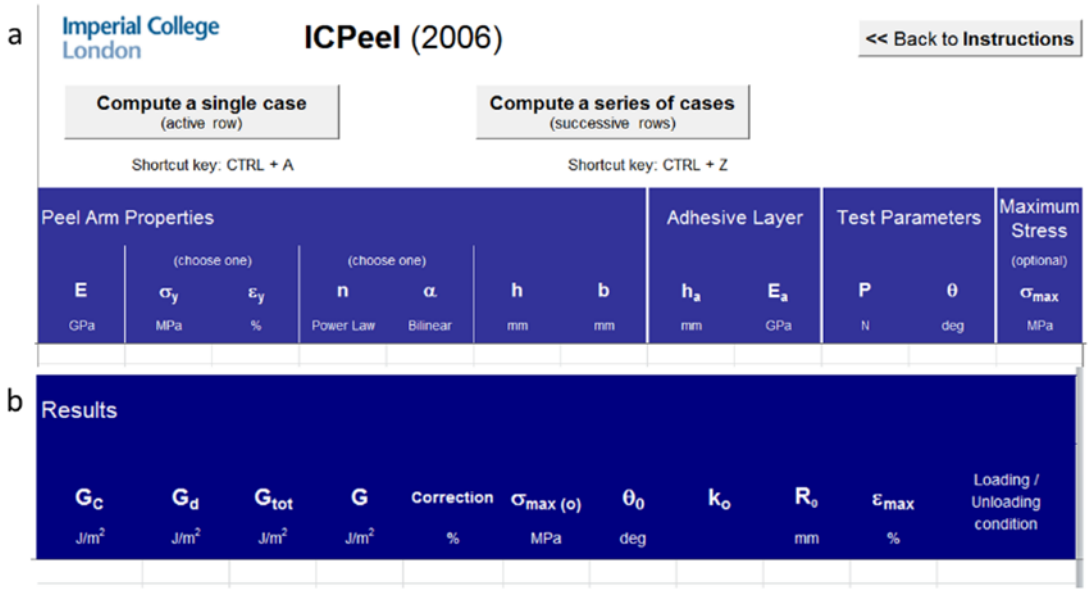


Figure 3.45: Interface of the ICPeel software (Zhang 2015)

For the peel arm properties, the power law model was selected for the analysis as the fracture energy for this model is lower than the bi-linear model for about 5% (Zhang 2015). The parameters of the peel arm properties are listed in Table 3.2.

Table 3.2: Peel arm properties

Parameter	Value
Low strain modulus (E_1)	61.0 GPa
High strain modulus (E_2)	1.16 GPa
Yields strain (ε_y)	0.044%
A (E_1/E_2)	0.019
Yield stress (σ_y)	26.84 MPa

3.4.4.2 Moisture Conditioning

In order to introduce the moisture to the sample, the post-bond moisture conditioning was conducted. This type of conditioning allowed the water to permeate simultaneously into the aggregate and bitumen. As shown in Figure 3.46, the whole sample was submerged into water for 14 and 28 days. After removing the sample from water bath, the sample should be tested within 1 hour.



Figure 3.46: The moisture conditioning of peel test sample

3.5 Asphalt Mixture Manufacturing

3.5.1 Binder Content

For the study mixture, only higher percentage of RWP-modified binders namely 7% is chosen for manufacturing asphalt mixture due to the most promising result in terms of its binder performance, as discussed in Chapter 5 and thus, allowed the best performance of asphalt mixture. For conventional mixture, two different penetration bitumen grades were used namely the control binder with penetration grade of 40/70 and SBS-modified binder. The control binder represents the similar penetration grade for 7% RWP-modified binders and the SBS-modified binder is the ready-made binder obtained from the supplier.

In term of the binder content, by Airey (2004) used 5.35% for producing asphalt mixture with polymer-modified binder (PMB) while Pareek et al. (2012)

reported that the optimum binder content using PMB is 5.02%. Thus, in this study, two binder contents were used in this study namely 5% and 6% by mass of the total mixture for comparison of the effect of different binder content.

3.5.2 Aggregate

The aggregate used in this study is Granite aggregate obtained from Bardon Hill Quarry, UK while the filler used is crushed limestone, which was obtained from Tunstead Quarry, Derbyshire, UK. For the asphalt mixture design, the 14 mm dense graded asphalt concrete according to BS 13108-1:2006 were used in this study for all asphalt mixtures and the gradation curve is shown in Figure 3.47. The target air void content for this design is 4%.

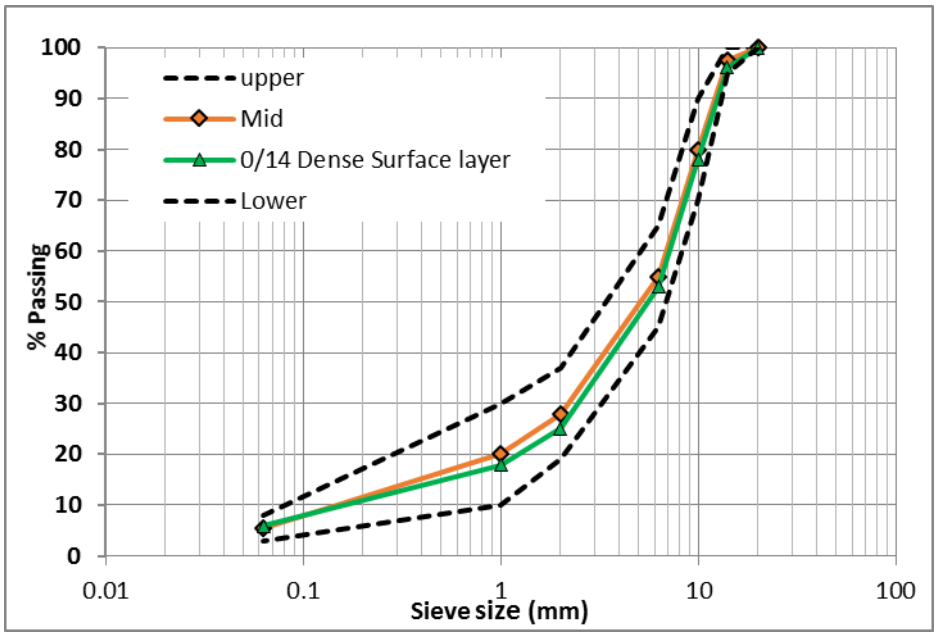


Figure 3.47: The 14 mm gradation curve

3.5.3 Mixing and Compaction

During the production of asphalt mixture, the procedures done for the mixing and compaction is according to the BS EN 12697-35: 2004 and BS EN 12697-33: 2003 respectively. The mixing and compaction temperature for asphalt mixture control binder were specified based on the viscosity of the binder in which according to the

Asphalt Institute, the temperature corresponding to the binder viscosities value of 170±20 mPa.s and 280±30mPa.s were chosen as mixing and compaction temperatures, respectively. While for 7% RWP-modified binders, this method could be applied due to higher mixing and compaction temperature for the mention viscosities which is higher than 200^{o}C for mixing. The higher mixing and compaction temperature are impractical in term of the workers' health as it would release hazardous fumes. Subhy (2017a) suggest that the mixing and compaction temperature were selected based on the sufficiency of the binder to coat the aggregate and the practicality to achieve the prescribed void contents. Therefore, the mixing and compaction temperature were specified as 190±5 and 170±5, respectively for manufacturing asphalt mixture containing 7% RWP-modified binders.

All aggregate and binders were preheated before mixing at specified mixing temperature for 8 hours and 3 hours, respectively. The aggregate and binder were mixed in a mechanical asphalt mixer as shown in Figure 3.48. The modified binder should be stirrer prior to pouring into the mixer to ensure that the RWP is uniformly distributed in the base binder. After uniformly mixed, the mixture were placed in a preheated slab mould (306 x 306 mm) and compacted by means of the compactor as shown in Figure 3.49 until the required slab thickness is achieved. The slab is then allowed to dry out at room temperature before core it with the dimension of 100 mm diameter and 40 mm thickness.



Figure 3.49: Mixing in the mechanical mixer



Figure 3.48: Steel roller

3.6 Asphalt Mixture Tests

3.6.1 Stiffness

The capability of asphalt mixture layer to spread the load applied can be accessed via stiffness modulus. This parameter is considered as a vital property of the asphalt mixture (Sreeram et al. 2018). The stiff mixture could spread the traffic loading over a wider area and vice versa (Subhy 2017a). The Indirect Tensile Stiffness Modulus (ITSM) test could be used to evaluate the stiffness modulus of the asphalt mixture and this test was carried out in accordance to BS EN 12697-26:2004 and the test involved is non-destructive test method. The Nottingham Asphalt Tester (NAT) machine was utilised for conducting this test in deformation-controlled mode and the configuration of the test is shown in Figure 3.50 for the indirect tensile mode. During the test, the compressive haversine load pulses were applied on the cylindrical specimen placed vertically and the two linear variable differential transducers (LVDTs) that placed opposite each other along diametrical section of specimen is used

to measure the deformation of specimen (Moghaddam et al. 2014). Cause of this test is non-destructive, the measurement was performed twice for each specimen in which after applying the first 5 loads cycles, the specimen was rotated for 90^{o} and the second 5 loads cycles was applied. Then, the ITSM result was obtained from the average of these two measurements. The stiffness modulus was calculated according to the following formula:

$$S = \frac{F\ x\ (\upsilon + 0.27)}{z\ x\ h} \qquad \text{Equation 3.34}$$

Where: F = applied load (N), υ = Poisson's ratio, z = horizontal deformation (mm), h = thickness of specimen (mm).

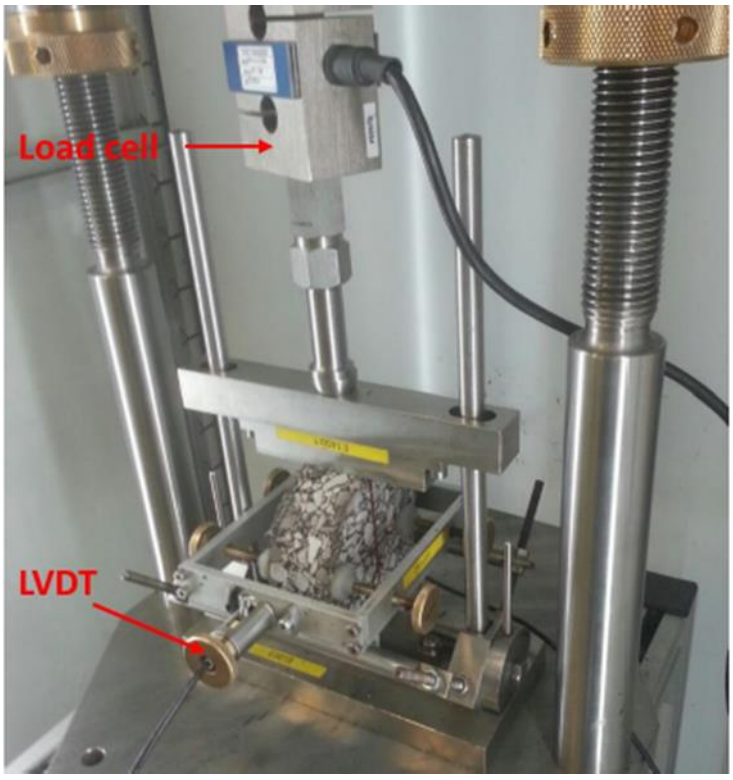


Figure 3.50: The configuration of ITSM test in the NAT machine (Subhy 2017)

The parameters were used as followed to perform ITSM test:

- Test temperature: 20 ± 0.5 ^{o}C
- Rise time: 125 ± 10 ms (the duration between the origination of load pulse from zero to the maximum value)
- Deflection requirements: 5 ± 2 µm
- Pulse durations: 3 s
- Number of conditioning pulses: 10
- Number of test pulses: 5
- Poison's ratio: 0.35
- Conditioning hour: at least 4 hours at the test temperature before testing

3.6.2 Fatigue

The fatigue behaviour of the asphalt mixture can be characterised by performing the Indirect Tensile Fatigue Test (ITFT) by means of NAT machine and the test is carried out according to the BS DD ABR (1995). Before conducting the ITFT test, the indirect tensile strength modulus parameter was obtained for each specimen at the specific fatigue stress level. This tensile strength value is used for calculating the initial strain of the specimen for constructing the fatigue line. Then, for ITFT, a cylindrical specimen is subjected to the repeated compressive loading across the vertical diametrical plane. The vertical deformation will appear and measure by the LVDTs that is built into the top part of the machine. The ITFT test configuration is shown in Figure 3.51 and conducted by using the following parameters:

- Test temperature: 20^{o}C
- Loading condition: controlled stress
- Loading rise-time: 124 ms

- Failure indication: 10 mm vertical deformation or specimen fracture occurred

The typical raw data from ITFT is exhibited in Figure 3.52. In the ITFT test, the traffic passing over the pavement is simulated by the pulses repetition in which the repeated application of compressive load on the specimen resulted in the strain (Tabakovic et al. 2006). The tensile strain at the centre of the specimen is calculated using the following equation:

$$\varepsilon_i = \frac{\sigma\,(1+3\,v)}{S}\ x\ 1000 \qquad \text{Equation 3.35}$$

Where:

ε_i = the initial strain in the centre of the specimen

v = Poisson's ratio (0.35)

σ = stress applied during the test.

The initial strain is a vital parameter in order to attain the fatigue line of the specimen as shown in Figure 3.53. By applying the linear regression of the data points, the fatigue life of the asphalt mixture can be characterised by the following relationship:

$$N_f = a(\varepsilon_i)^b \qquad \text{Equation 3.36}$$

Where:

a and b = fitted parameters

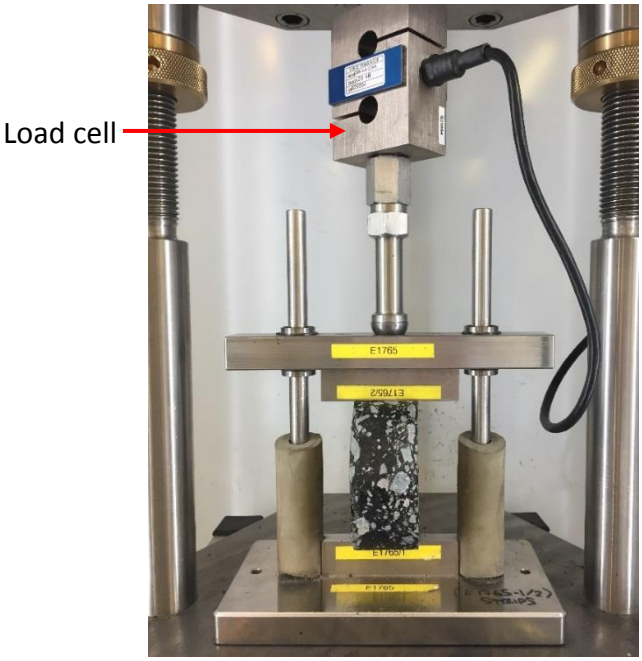


Figure 3.51: The configuration of ITFT test on NAT machine

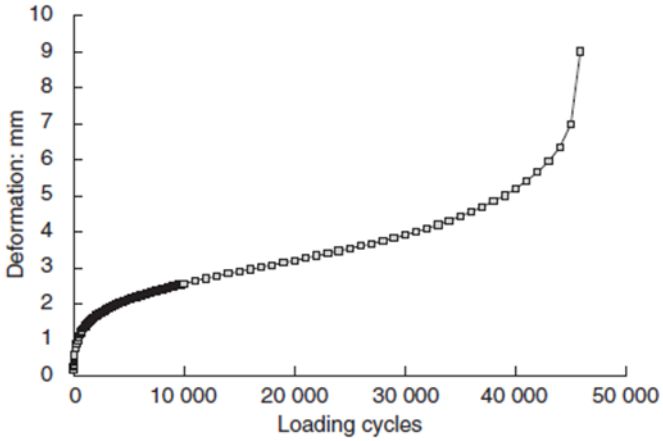


Figure 3.52: Typical raw data obtained from the ITFT (Cocurullo et al. 2008)

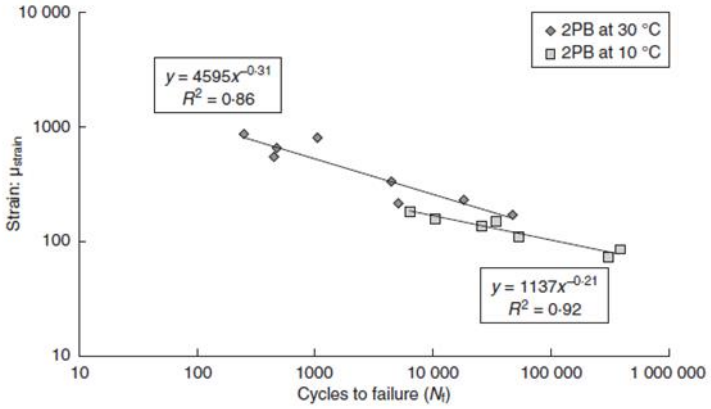


Figure 3.53: Example of the fatigue line of asphalt mixture (Cocurullo et al. 2008)

3.6.3 Rutting

In order to characterise the rutting behaviour of the asphalt mixture, the Repeated Load Axial Test (RLAT) is used. The RLAT was performed on cylindrical specimen and conducted in accordance to standard procedure as specified in BS DD 226 by means of Nottingham Asphalt Tester (NAT) machine. The specimen used is in the cylindrical form with a diameter of 100 mm and thickness around 40 mm. Before being tested, the specimen that contains two faces are coated with a thin layer of blend of silicone grease and graphite powder. In this test, the specimen was vertically positioned between the upper and lower steel loading platen and the configuration of the test is shown in Figure 3.54 The unconfined axial stress is applied vertically by the load actuator on the specimen for a pulse that consist of 1 second of load application and 1 second of rest period.

The RLAT condition and parameters used in this study are as follows:

- Test temperature: 40^{o}C
- Test duration: 10800 seconds (5400 pulses)
- Axial stress: 100 kPa
- Conditioning stress: 10 kPa for 600 seconds

- Conditioning hour: at least 4 hours at the test temperature before testing
- Repetition: three specimens for each mixture and the average values are reported

The raw data obtained from the RLAT is shown in Figure 3.55. This plot generally gives three distinctive phase of the material as demonstrated by Figure 3.56 which consists of primary zone (as the loading cycle increased, the deformation rate is decrease but the vertical strain accumulates rapidly due to the combination of adjustment of loading platens and densification of material) secondary zone (the rate of deformation of the mixture is constant with loading cycle due to the strain increase proportional to the load cycle and the steady state response is achieved in this zone) and tertiary phase (the vertical strain accumulates rapidly again, thus increase the deformation rate with loading cycles which indicates sample failure has occurred) (Zhou & Scullion 2002; Zhou & Scullion 2003; Zhou et al. 2004; Zhang et al. 2013; Subhy 2017a). Widyatmoko (2008) stated that if the axial strain of the asphalt mixture is lower than 2%, thus better rutting performance is exhibited by this mixture. Besides that, Gibb (1998) and Airey (2004) suggest to use the minimum strain rate ($\mu\varepsilon/cycle$) because this parameter is not effected by the initial strain during the test as the calculation of this parameter involve only at the steady state phase. In this study, the slope of steady state phase is obtained between pulses of 3000 to 5000 and the minimum strain rate is calculated as follows:

$$Minimum\ strain\ rate\ ((\mu\varepsilon/cycle) = \frac{\varepsilon_{5000}-\varepsilon_{3000}}{2000}\ x\ 10^{-6} \quad \text{Equation 3.36}$$

Where:

ε_{5000} = accumulated strain at 5000

ε_{3000} = accumulated strain at 3000

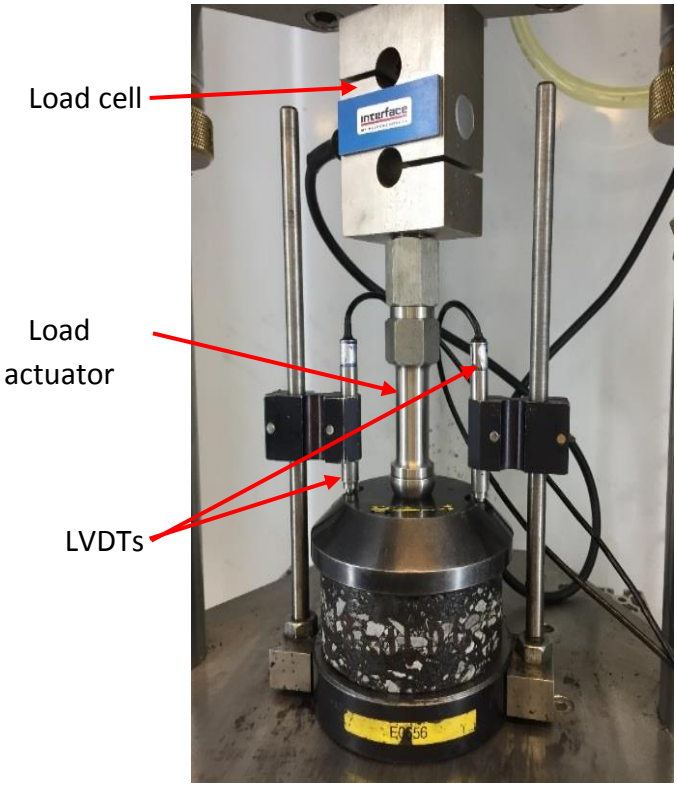


Figure 3.54: Configuration of RLAT test in NAT

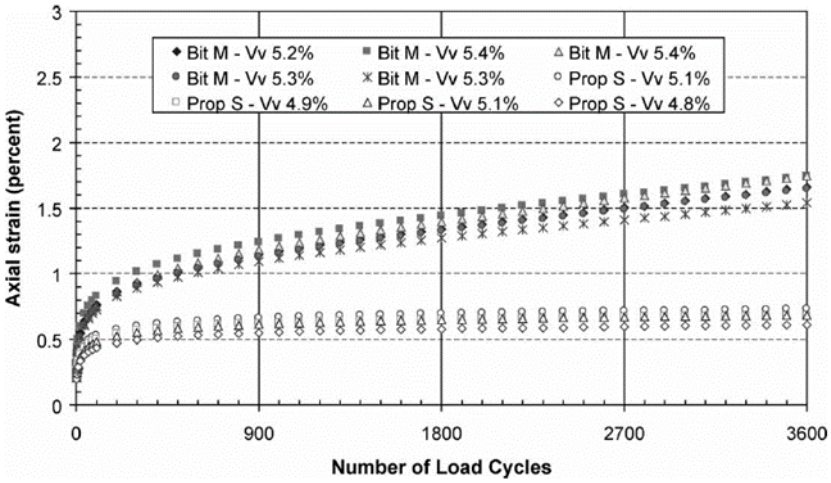


Figure 3.55: Typical raw data of asphalt mixture from RLAT (Airey 2004)

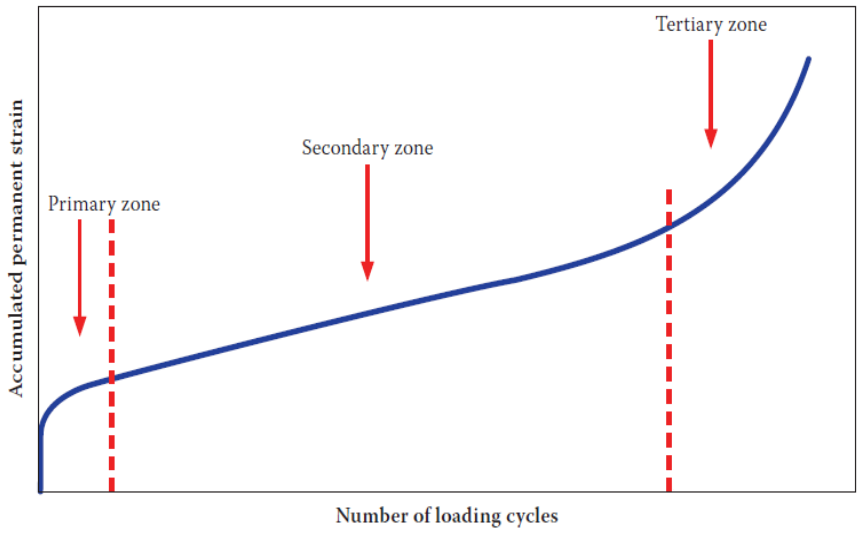


Figure 3.56: Typical three-stage curve of accumulated permanent strain versus the number of loading cycles (Walubita et al. 2013)

3.7 Summary

The methodology and material used in this study has been outlined in this chapter. Six type of recycled plastics was used for modifying 160/200 penetration grade bitumen and later, two different type of binders which are 40/60 penetration grade bitumen and SBS-PMB were used for further comparison in terms of its properties. For the asphalt mixture, the 14 mm dense graded asphalt concrete has been selected with aggregate and filler used were granite and limestone, respectively. The testing programme was divided into three parts namely the preparation of recycled plastic modified bitumen, binder tests and asphalt mixture tests. The preparation of recycled plastic modified bitumen involves identification of potential recycled plastics and optimisation of blending parameter. The assessment of modified bitumen at binder tests includes four properties namely rheological, chemical, structural and performance have been used. For asphalt mixture test, three tests have been conducted including stiffness test (indirect tensile stiffness modulus test), fatigue test (indirect tensile fatigue test) and rutting (repeated load axial test).

CHAPTER 4: SELECTION AND INCORPORATION OF RECYCLED WASTE PLASTIC (RWP) IN BITUMEN

4.1 Introduction

The main objective of this chapter is to identify the potential of RWP to be used as a modifier in bitumen. Then, the most promising and appropriate blending condition of RWP-modified binder that is qualified to be used for further binder testing that will be carried out in the next chapter.

In order to obtain the suitable RWP for modifying the base binder, the melting point of the RWP is obtained by means of Differential Scanning Calorimetry (DSC) test. Then, the blending parameter which are temperature, time and shear rate were optimised by using mixer. Lastly, the chemical and structural characterisations were examined for the effect of ageing during blending by means of Flourier-transform Infrared (FTIR) spectroscopy and stability of the RWP-modified binder during high temperature storage by using Dynamic Shear rheometer, respectively.

4.2 RWP Screening

The screening of the RPWs were done based on the thermal analysis results from the DSC test and the remaining RPWs will be undergo blending process. The possibility of incorporating all RWPs for bitumen modification was explored. The RWP that has melting point below than 200^{o}C were considered suitable for modifying bitumen because the bitumen will experience oxidation when exposed to the high temperature for long time (Pérez-Lepe et al. 2003) that later may lead to the polymer

degradation (Larsen et al. 2009). The 200°C was used as a maximum blending temperature as recommended by Al-Dubabe et al. (1998). Besides that, Casey et al. (2008) have found that mixing of polymer that having high melting points is impractical to make. If the temperature of RWP obtained from the DSC test is within the range of allowable blending temperature with base bitumen (<200°C), the RWP will be used and the selected blending temperature must be beyond this temperature. The example of output test of DSC is shown in Figure 4.1 and Figure 4.2 and the melting point for each RWP are shown in Table 4.1 (value rounded to the nearest). As seen in this table, it was clear that PET, PS and PVC were having melting point higher than suitable allowable temperature for blending with bitumen. Thus, these three RWPs were not included in the study for further tests and the remaining RWP which are LDPE, HDPE and PP were qualified to modify bitumen.

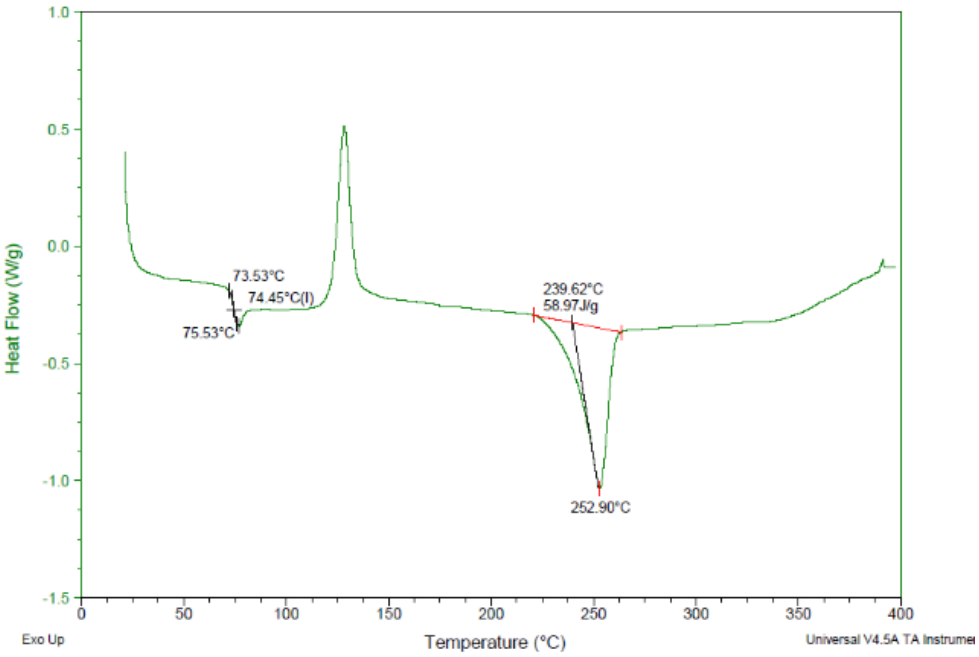


Figure 4.1: DSC result of recycled PET

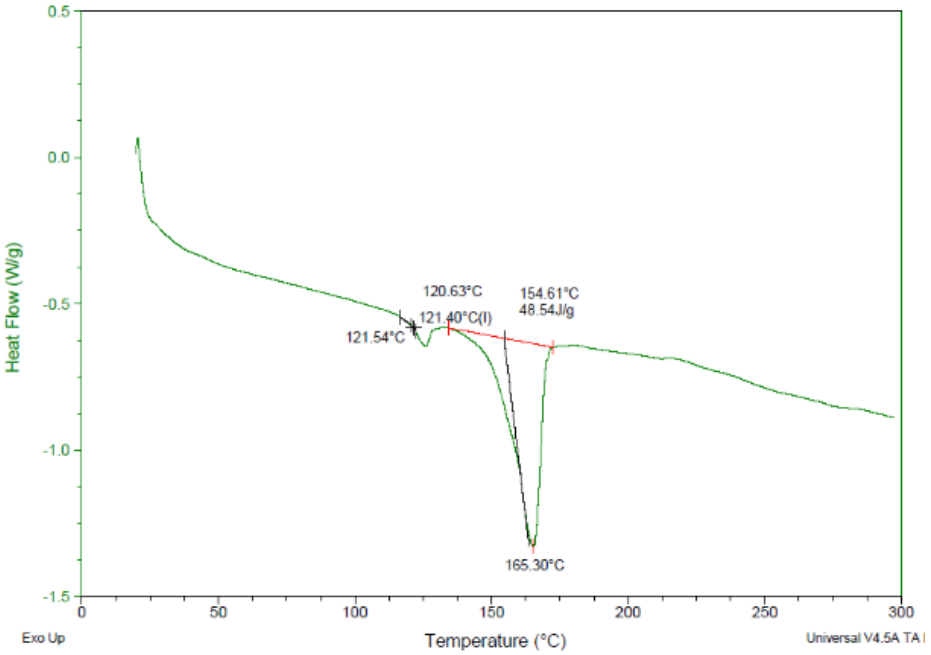


Figure 4.2: DSC result of recycled PP

Table 4.1: Melting point and Blending temperature of RWPs

RWP	Melting Point (°C)
PET	253
HDPE	134
PVC	>300
LDPE	125
PP	165
PS	>300

4.3 Bitumen Modification



Figure 4.4: Equipment for blending



Figure 4.3: Equipment for blending

The blending code for each RWP used during optimisation process is summarised in Table 4.2. Only 3% modification is carried out for optimising purpose because the amount of RWP obtained from the supplier is minimal. Differing in blending time and temperature, all modified binders were tested for the softening point in order to obtain a dispersive mixing of RWP in base bitumen. The blending of RWP and base

bitumen were prepared by using the shear mixer and stirrer shown in Figure 4.3 and Figure 4.4, respectively. The blending was performed by applying a constant low shear rate of 250 rpm. The reason of using low shearing rate for obtaining modified binder because Al-Dubabe et al. (1998) stated that some polymer only required low shear rate of mixing to well disperse into the base bitumen. Moreover, blending PMB with low shear speed for a few hours will allow the polymer to disperse into the blend and a homogeneous mixture of PMB will be produced (Hunter et al. 2015). Besides that, Saboo and Kumar (2016) investigated the optimum blending requirements of PMB and they found out that the homogeneous modified binder is obtained from the optimum blending parameter which is strongly dependent on the blending temperature followed by blending time and then shear rate.

Two different mixing temperatures were chosen that is from 150^{o}C to 190^{o}C. The mixing temperature were chosen according to the melting temperature of each RWP. In details, LDPE and HDPE have melting point below 150^{o}C, and hence 150^{o}C and other additional of 20^{o}C were used as proposed mixing temperature. Same procedure has been applied for PP and the mixing temperature used are 170^{o}C and 190^{o}C.

At first, 3 hours of modification blending were carried out and in order to optimise the time of blending parameters, in every 30 minutes intervals, a sample of binder is taken and tested for softening point test. The softening point is the recommended criterion for defining the homogeneity because the softening point defines the beginning of fluidity of binder, which identifies the critical point in the physical state of bitumen and softening point has good correlation with rutting (Al-Dubabe et al. 1998) . The optimum time of blending were obtained once the softening

point stabilises and no significant increase at the particular blending temperature was recorded. While blending temperature will be identified by the highest softening point value of PMB recorded among tested temperatures. Increase in softening point means that the modified bitumen will be less susceptible to temperature changes. This indicates that the resistance of the binder to the effect of heat is increased, the tendency to soften during hot weather will reduce and the rate of rutting will decrease due to the increase in softening point (Mahrez & Karim 2010).

Table 4.2: Blending Code

Recycled Waste Plastic (RWP)		Mixing Time (Minutes)	Mixing emperature (°C)	Blending Code
Type	Percentage			
LDPE	3	0-180	150	LDPE-3-150
LDPE	3	0-180	170	LDPE-3-170
HDPE	3	0-180	150	HDPE-3-150
HDPE	3	0-180	170	HDPE-3-170
PP	3	0-180	170	PP-3-170
PP	3	0-180	190	PP-3-190

4.4 Blending Protocol

1. The process began by heating a can containing 200 gram of base bitumen in the oven at tested temperature until it became fluid.
2. After the base bitumen is completely melted, the can was put on a hot plate and low shear mixing was applied at speed of 250 rpm. The stirrer is totally immersed into the hot base bitumen during the blending and heat the can until the target temperature is reached.

3. When the target temperature is reached, the modifier is slowly added to the base bitumen and the blending time started. A thermometer was used to monitor the mixture's temperature.
4. During the blending process, every 30 minutes intervals from the beginning of the test, about 10 grams of binder were tested for softening point test to find the optimum blending time and temperature. The blending was continued for up to 180 minutes for each RWP.

4.5 Optimisation of Blending Duration

The variation of the softening point with time for 3% of RWP in the base bitumen is shown in Figure 4.5 and the blending parameter used in the blending procedure is summarised in Table 4.2. As can be observed, at the beginning, HDPE modified binder showed a little decrease after 60 minutes of blending but it appeared to increase after that and overall, all RWP modified binders displayed a similar trend of softening point value profile during the blending process. Increased in softening point value was presented by all RWP modified binder blends up to blending time of 120 minute and then, it showed a decrease and relatively uniform after about 120 minutes of shearing, meaning that the RWP has already been dispersed thoroughly and extended blending time will not affect the morphology of the blend (Dalhat & Al-Abdul Wahhab 2015). As the test temperature increased, LDPE and HDPE modified binder blends have higher softening point value while PP modified binder blend showed vice versa. Based on the observation trend in Figure 4.5, the highest softening point value is desirable to perform the best modified binder blend that is beneficial to the pavement and thus, the optimum blending time and temperature of

RWP LDPE, HDPE and PP modified binder was 120 minutes and 170°C, respectively. Moreover, the blending temperature obtained from this study was parallel with the previous studies for virgin plastic which is in the range 160 to 180°C (Al-Dubabe et al. 1998; Wahhab & Abaker 2004; Polacco et al. 2005; Al-Hadidy & Yi-Qiu 2009c; Attaelmanan et al. 2011; Habib et al. 2011). This indicates that recycled material may have similar properties with the virgin material.

Then, the base binder with penetration grade of 160/220 was modified with 3, 5, 7 and 9% of LDPE, HDPE and PP using the blending parameter obtained above. Unfortunately, the blend containing 9% of RWP could not be done because it appears to be fully saturated and reduce in workability. Furthermore, the 9% RWP blends were discarded from the study.

Table 4.3: Optimum blending times for each RWP

RWP	Melting Point (°C)	Blending Temperature (°C)
PET	253	-
HDPE	134	170
PVC	>>300	-
LDPE	125	170
PP	165	170
PS	>>300	-

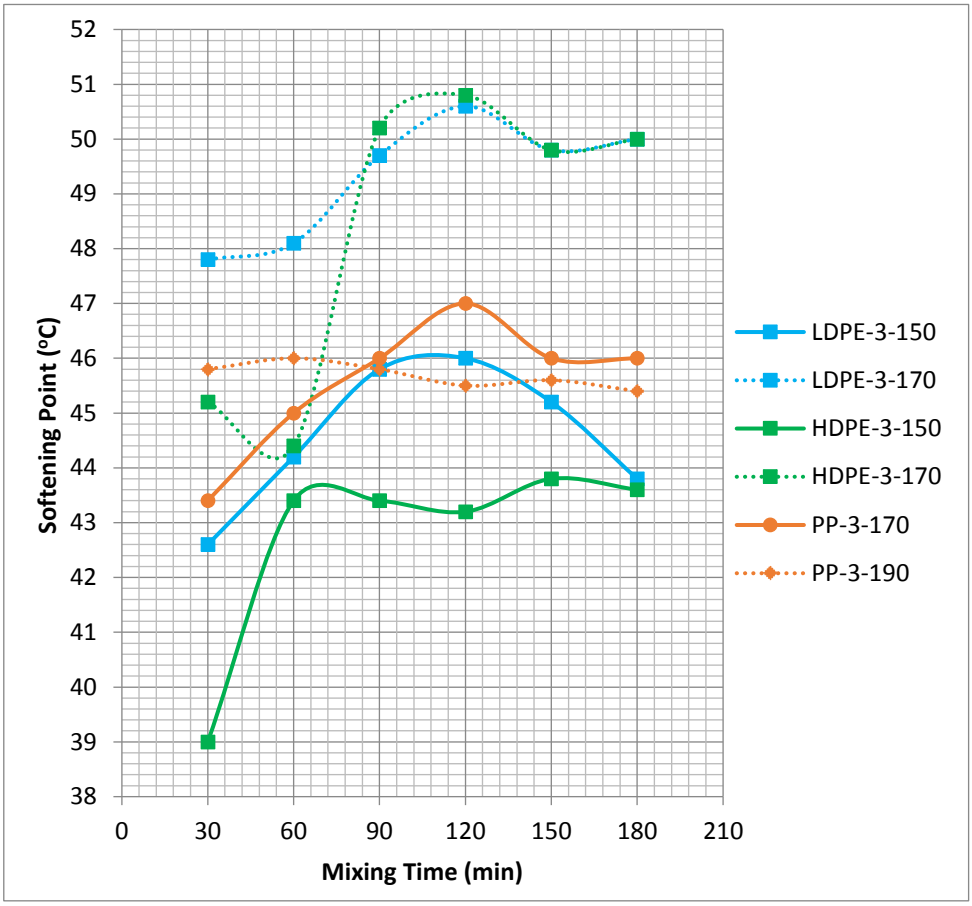


Figure 4.5: Softening point value of RWP-modified binders at different blending times and temperature

4.6 Chemical Characteristics

The ageing effect during blending was analysed by means of FTIR test and conducted as described in Section 3.3.1.3. In this analysis, RWP-modified binders were used as a sample to assess the ageing effect because these binders were produced in the laboratory by means of heating for 2 hours at high temperature and then, the comparison has been made with base binder. The exposure on the heat for a prolonged period of time at high temperature could age the binder in which it will make the binder more harden due to the oxidation and volatilisation of the light components of the binder.

4.6.1 Effect of Blending Process on Ageing

In this study, the FTIR spectrometer was used to analyse the evolution of chemical structures of the RWP-modified binders after undergoing blending process at temperature of 170^{o}C for 2 hours with shear rate of 250 rpm. The carbonyl (C=O) and sulfoxide (S=O) were used to evaluate the effect of ageing on bitumen during production of modified binder. Ageing occurs when the formation of carbonyl and sulfoxide groups of RWP-modified binders are higher than the base binder.

The FTIR spectra of the base bitumen and all RWP-modified binders are given in Figure 4.6. The area of absorbance bands of carbonyl and sulfoxide group is displayed in Figure 4.7 (a) and (b), respectively were analyses and calculated in term of its structural indices. It can be observed that the highest carbonyl area is displayed by the base binder in comparison with other RWP-modified binders while some RWP-modified binders demonstrate similar carbonyl area. For the of sulfoxide group area, a significant discrepancy can be noticed between the base binder and other RWP-modified binders. This indicates that oxidation occurs during the blending process. However, the differences of absorbance band area for carbonyl is difficult to distinguish among RWP-modified binders as the spectra are overlapping and thus, the index of carbonyl and sulfoxide is calculated and exhibited in Figure 4.8 for comparison. A high value of functional group index represents a formation of that functional group is higher and thus, ageing occurs during the production of the binder (Wu et al. 2009). As shown in Figure 4.8, after being blending at high temperatures for 3 hours, the indices of carbonyl group for RWP-modified binders experience a significant reduction in comparison with base binder while the sulfoxide indices

demonstrate an increase from 0.0039 for the base binder to the minimum of 0.0066 for RWP-modified binders. This implies that the blending process of RWPs into base binder can lead to the less formation of the carbonyl groups but in contrary occurs to the sulfoxide groups. Besides that, the change of carbonyl and sulfoxide indices is almost similar for all percentages of RWP-modified binders (except for 3PP-modified binder) as shown in Figure 4.8. Therefore, it can be concluded that the addition of RWP into base binder that blended under a shear rate of 250 rpm at high temperature for 3 hours may increase the susceptibility of the binder to ageing due to the oxidation occurred during blending process as evidence of higher sulfoxide indices of the modified binders but might be insignificant as the carbonyl indices of RWP-modified binders is lower than the base binder.

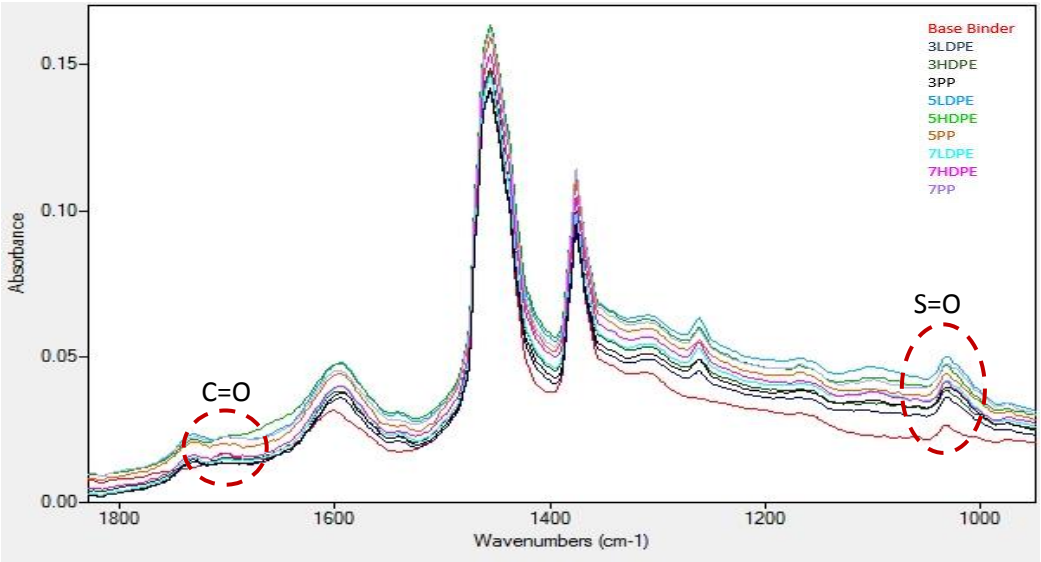


Figure 4.6: FTIR Spectra of different binders

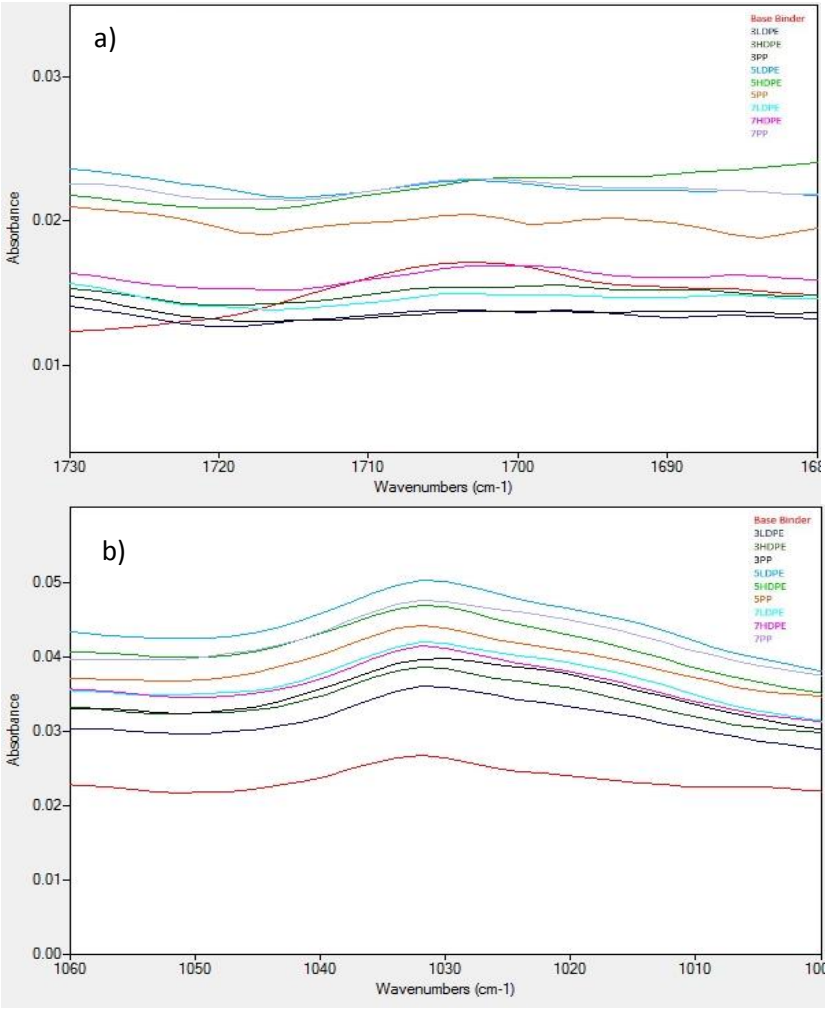
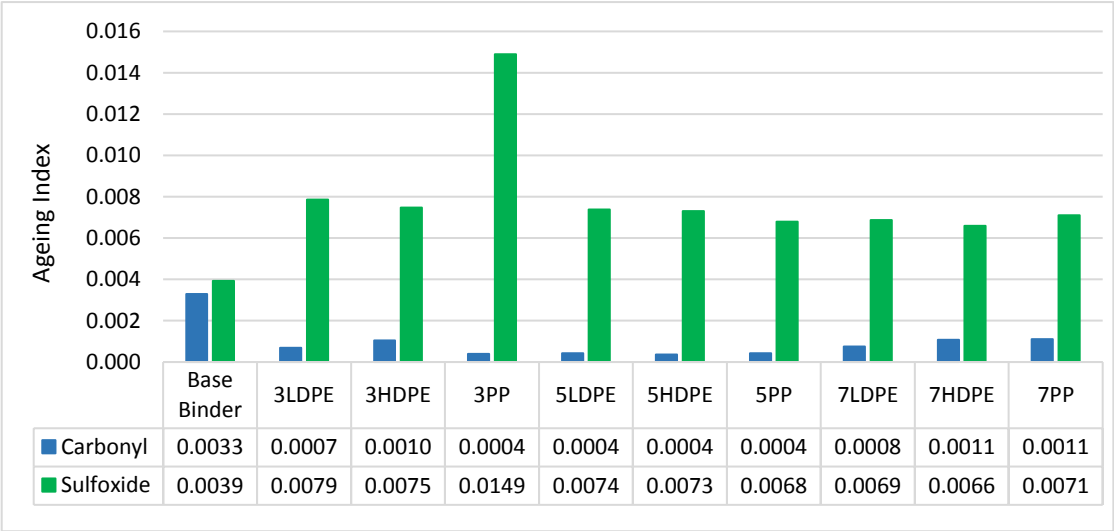


Figure 4.7: FTIR Spectra of different binders for a) Carbonyl region, b) Sulfoxide region



	Base Binder	3LDPE	3HDPE	3PP	5LDPE	5HDPE	5PP	7LDPE	7HDPE	7PP
Carbonyl	0.0033	0.0007	0.0010	0.0004	0.0004	0.0004	0.0004	0.0008	0.0011	0.0011
Sulfoxide	0.0039	0.0079	0.0075	0.0149	0.0074	0.0073	0.0068	0.0069	0.0066	0.0071

Figure 4.8: Carbonyl and sulfoxide ageing indices for different binders

4.7 Structural Characteristics

The changes in the structure of RWP-modified binders at high temperature storage was assessed by the storage stability test and performed as described in Section 3.3.2.1. For evaluating the stability of the binder at a higher temperature, the comparison of RWP-modified binders and SBS-modified binder were made in which the SBS-modified binder and RWP-modified binders were manufactured by the supplier and in the laboratory, respectively. The frequency sweep tests were carried out to evaluate the complex modulus of binder for different amount of RWP added at 72 hours of storage condition. To analyse this viscoelastic property, a plot of isotherm curve for top and bottom sample of the binder at temperature of 50^{o}C is provided in Figures 4.9, 4.10, 4.11 and 4.12.

4.7.1 Effect of Stability of Polymer at High Temperature Storage

The isotherm curves at frequency range of 0.1 to 10 Hz obviously illustrated the differences of the binder's behaviour upon storage for 72 hours as shown in Figures

4.9, 4.10 and 4.11. All figures clearly depicted that the RWP-modified binders studied are prone to separation as the curve of bottom and top sections of each RWP-modified binder showed significant differences. It can be seen that all curves displayed similar trend with significant variation in stiffness between bottom and top sections of the sample with the top of the section is stiffer compared to the bottom section. This means that the RWP tends to migrate to the top of the tube during separation. The variation in stiffness is expected when longer storage time used (Zani et al. 2017).

However, SBS-modified binder isotherm curves displayed in Figure 4.12 exhibited good resistance to storage after 72 hours with no significant discrepancy appeared for complex modulus of top and bottom section. Meaning that the SBS-modified bitumen samples is stable. It should be mention here that the SBS-modified binder was provided by a supplier in 'as-sold' condition. Hence, the quality of this binder is controlled. So, the good stability of the SBS-modified binder at high temperature storage is expected.

It has been reported by Garcia-Morales et al. (2007) that the addition of waste polymer could enhance the mechanical properties of the binder at low and high temperature although the storage of this modified binders at high temperature was unstable.

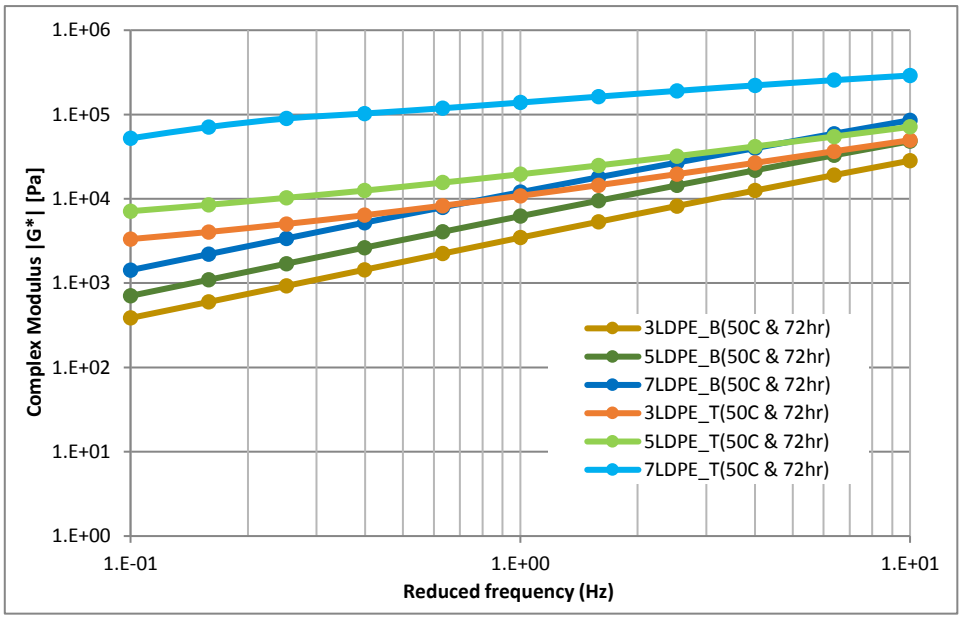


Figure 4.10: Isothermal plots for LDPE-modified bitumen at 50°C after 72 hours of storage

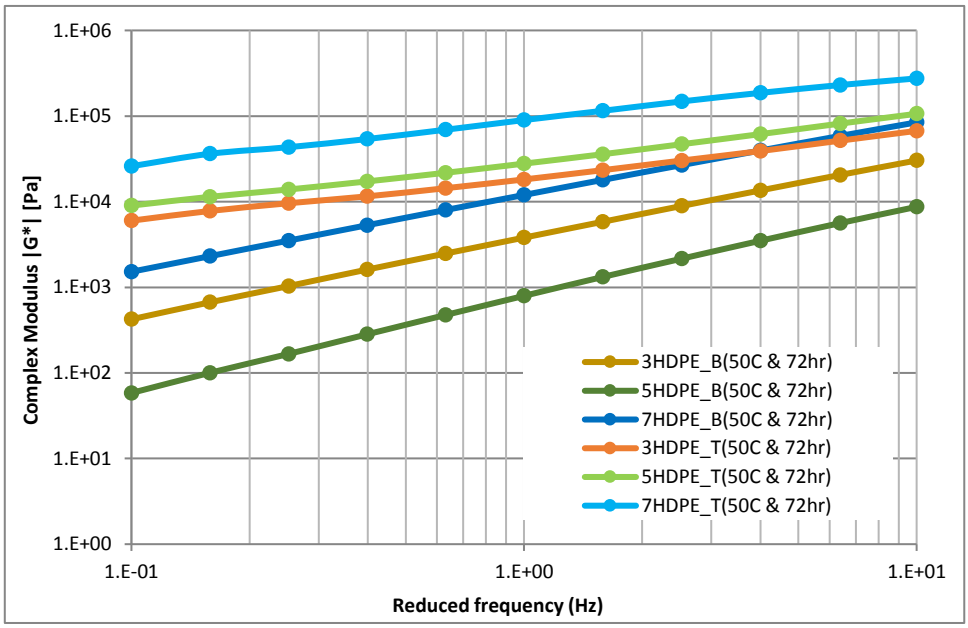


Figure 4.9: Isothermal plots for HDPE-modified bitumen at 50°C after 72 hours of storage

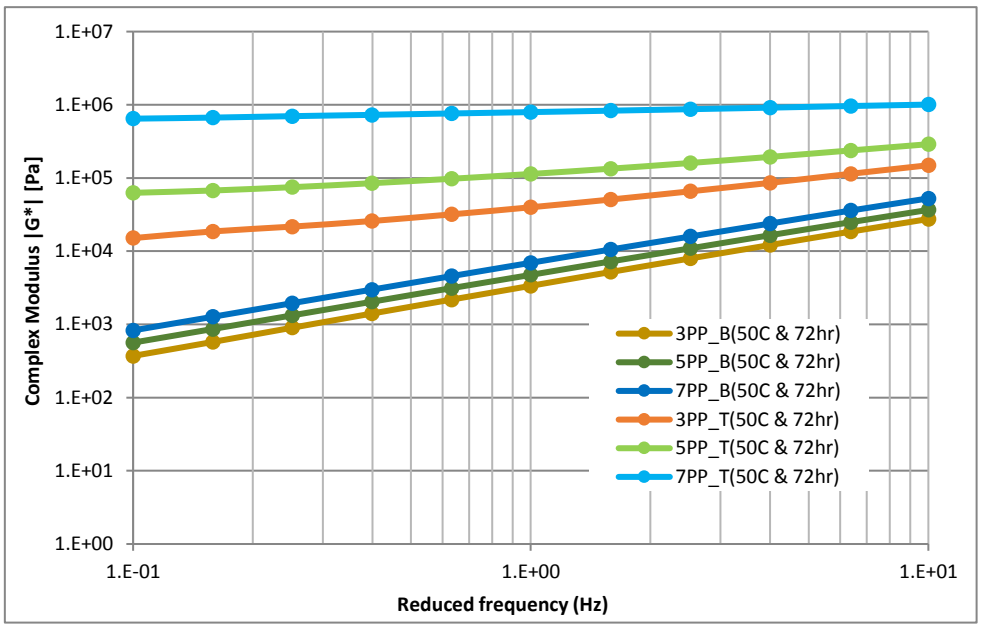


Figure 4.12: Isothermal plots for PP-modified bitumen at 50°C after 72 hours of storage

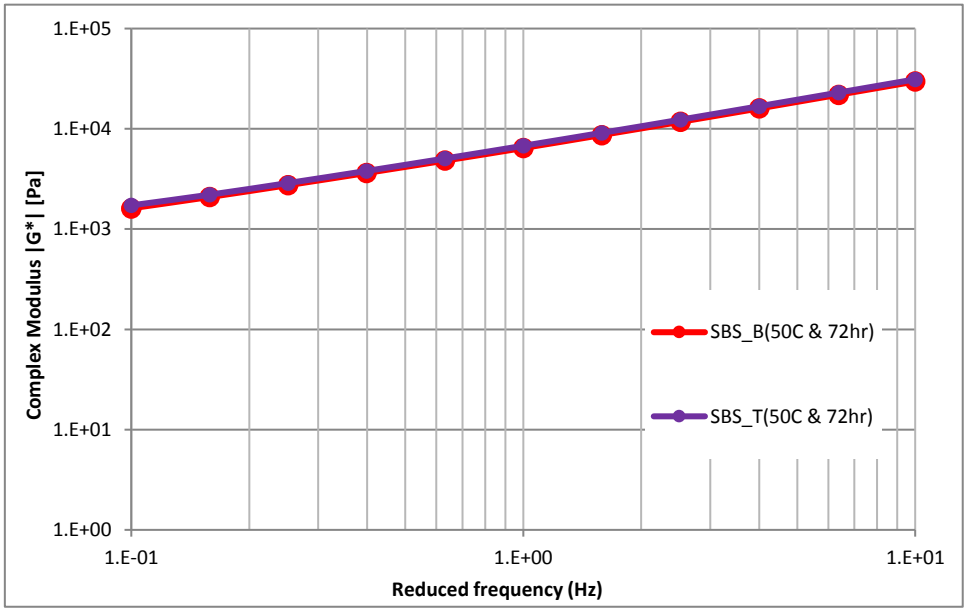


Figure 4.11: Isothermal plots for SBS-modified bitumen at 50°C after 72 hours of storage

4.8 Summary

This chapter described the procedures that were used for incorporating RWP in base bitumen. Based on the DSC test for obtaining the melting point of six RWPs, only three RWPs were qualified to be used as a modifier which is LDPE, HDPE and PP due

to the melting point of these RWPs are below 200^{o}C while the other RWPs namely PET, PS and PVC have melting point more than 200^{o}C. The blending of RWP and base binder were carried out by applying a constant low shear rate which is 250 rpm with the optimum blending time and temperature of RWP for LDPE, HDPE and PP modified binder are 120 minutes and 170^{o}C, respectively.

1. Chemical analysis:
    - The effect of ageing during production was examined by means of FTIR showed that the carbonyl band area of base binder is highest in comparison with all RWP-modified binders while these RWP-modified binders demonstrate higher area of sulfoxide band than the base binder.
    - The addition of RWPs into base binder by using blending parameter obtained earlier may lead to the oxidation and thus, increase the ageing susceptibility of the modified binders but might be insignificant due to the lower carbonyl indices of these binders when compared with the base binder.
2. Structural analysis:
    - The storage stability of RWP-modified binders at higher temperature was evaluated and compared with 'as-sold' condition bitumen which is SBS-modified binder. As expected, the SBS- modified binder showed good stability at high temperature storage while the RWP-modified binders for all modification prone to phase separation.

CHAPTER 5: **RWP-MODIFIED BINDER CHARACTERISTICS**

5.1 Introduction

From the previous chapter, it was found that only three RWP were suitable to modify the base binder consisting of LDPE, HDPE and PP. For each RWP, samples of modified binder were prepared by using the RWP at 3, 5 and 7% of bitumen weight.

In this chapter, the performance-related properties of the modified binders were investigated and then compared with the unmodified binder. For this purpose, conventional tests have been carried out for characterising the basic properties (penetration, softening point, penetration index and viscosity) of the RWP-modified binder followed by the performance properties in term of fatigue cracking, fracture resistance and rutting. The details of the results are presented in this chapter. Lastly, the most promising percentage of RWP-modified binders for each RWP in terms of improvement were selected for further experimental study in the next chapter.

5.2 Conventional Tests

The conventional tests, encompassing penetration, softening point and viscosity tests, were performed to evaluate the consistency or hardness of the binders. Besides that, the temperature susceptibility of the binders was assessed by means of the penetration index (PI).

5.2.1 Penetration and Softening Point

The control and modified binders were subjected to penetration and softening point test to obtain their conventional properties in accordance with European standards

which are EN 1426 2015 and EN 1427 2015, respectively. Yu et al. (2015) stated that the penetration value is a depiction of binder consistency which reflects its rheological properties and the softening point represents the plastic flow of the binder which is related to high temperature stability. Table 5.1 summarises the results of penetration and softening point of all the binders. It can be seen that the addition of RWP resulted in a decrease in penetration which indicates that the modification of base binder with RWP tends to increase the hardening of the binder regardless of the type of thermoplastics added. Moreover, the effect of increasing RWP concentration led to a reduction in penetration. This means consistency of the modified binders is increased and thus, demonstrates a high resistance to deformation. Unfortunately, hardening the binder may affect the flexibility of the bitumen and thus will affect the resistance to fatigue of the binder itself (Mahrez & Karim 2010).

For the softening point value, the modified binders showed higher values than the base binder and as the concentration of RWP added increased, the softening point value increased linearly. This is due to the hardening of the binder in which a higher temperature is needed to soften the binders as the binder became stiffer. Higher softening point values are favourable because they indicated that the addition of RWP enhanced the softening temperature of the binder which may improve its high temperature stability and make the binder less susceptible to permanent deformation.

Overall, the results show that the additional amounts of thermoplastic influence the conventional properties of the binders with the increase in

concentration of RWP added to modify the base binder leading to the penetration value decreasing, and the softening point values increasing. This implies that the higher level of modification of RWP modified binder could improve the high temperature performance of the pavement. From the conventional properties of the RWP-modified binders, there is the possibility of improved pavement performance characteristics in term of rutting, fatigue, and durability which can be further assessed with performance tests.

Table 5.1: Result of penetration and softening point test

Binder		Penetration (dmm)	Softening Point (°C)
Base Binder (BB)		181	37.4
40/60-Pen (Control Bitumen, CB)		47	50.4
SBS PMB		87	84.2
Polymer	**Modification (%)**	**Penetration (dmm)**	**Softening Point (°C)**
LDPE	3	105.3	47.8
	5	62.3	83.8
	7	42.2	104.5
HDPE	3	88.0	50.8
	5	56.4	80.6
	7	42.4	119.0
PP	3	96.6	46.4
	5	62.7	61.6
	7	44.5	120.0

5.2.2 Penetration Index (PI)

The sensitivity of the binders to temperature can be evaluated through the penetration index (PI). The temperature susceptibility of binder decreases if the PI value increases and the normal PI value for bitumen is ranging between -2 and +2. Binder that has PI value more than +2 have low susceptibility to temperature while excessively susceptible to high temperature if the PI value is less than -2. Asphalt mixtures containing binders with higher PI values (meaning binders with lower temperature susceptibility) are favourable as these asphalt mixtures have better resistance to cracking and rutting at low and high temperatures, respectively (Isacsson & Lu 1999). An evaluation of Table 5.2 showed that the modification of base bitumen with RWP resulted in an increase in PI values or in other words, the temperature susceptibility is improved in comparison with base binder. This indicates that the sensitivity of modified binders against temperature decreased. Therefore, the RWP modified binders can be used in a wider temperature range than the base binder (Köfteci et al. 2014). The PI value is approaching minimum value as the percentage of RWP added decrease, meaning the lower modification level have higher sensitivity to temperature compared with higher modification level. Previous studies have stated that the improvement of temperature susceptibility is expected for modified binders as it is one of the aims of modifying bitumen with polymer (Airey 1997; Isacsson & Lu 1999; Nassar et al. 2012).

Table 5.2: Penetration index of binders

Binder	PI
Base Binder (BB)	-1.69
40/60-Pen (Control Bitumen, CB)	-1.24
SBS-MB	6.54
3LDPE	0.23
5LDPE	5.45
7LDPE	6.64
3HDPE	0.49
5HDPE	4.74
7HDPE	7.88
3PP	-0.47
5PP	1.95
7PP	8.07

5.2.3 Viscosity

The flow properties regarding the pumpability and workability of the binder were determined by viscosity testing. Kalantar et al. (2009) described that if the viscosity of bitumen is too high, the binder may not be able to coat the aggregate completely in the asphalt mixture while if the viscosity is too low, binder drainage is likely to occur during the storage and transportation of the mix. The viscosity test was carried out using a Brookfield rotational viscometer and conducted in accordance with the procedure described in BS 13302-505:2010. The SHRP binder specification states that the viscosity of the bitumen must be less than 3 Pa.s at 135^{o}C for manufacturing of asphalt mixture using the binder (Jiménez del Barco Carrión 2017). The graph of rotational viscosity conducted over a temperature range of 90 to 200^{o}C on base binder and modified binders for different levels of modification as shown in Figure

5.1. It was found that all binders satisfy the SHRP requirement at 135^{o}C. However, the viscosity of all the RWP-MBs have higher viscosity values than the base binder.

From Figure 5.1, it can be seen that the viscosity values are higher at low temperatures and higher concentration of RWP-modified binders. Nevertheless, the binders become less viscous as the temperature is increased and the amount of RWP incorporated into the bitumen decreases. At the same level of modification, the binders tend to exhibit almost similar viscosity values regardless of the type of RWP. This means that the concentration of RWP used to modify the base binder has a greater effect on the viscosity rather than the type of RWP. Nassar et al. (2012) stated that the increase in the viscosity value at high temperatures is a good property for rutting resistance. Unfortunately, when the concentration of RWP incorporation into the base binder is increased, its workability is decreased as the binder becomes more viscous. This results in the need to increase the mixing and compaction temperatures for manufacturing. Many researchers have pointed out that an increase in energy consumption levels is undesirable (Köfteci et al. 2014). However, Casey et al. (2008) notes that most PMBs are typically mixed in asphalt mixture production at temperatures between 170^{o}C and 195^{o}C.

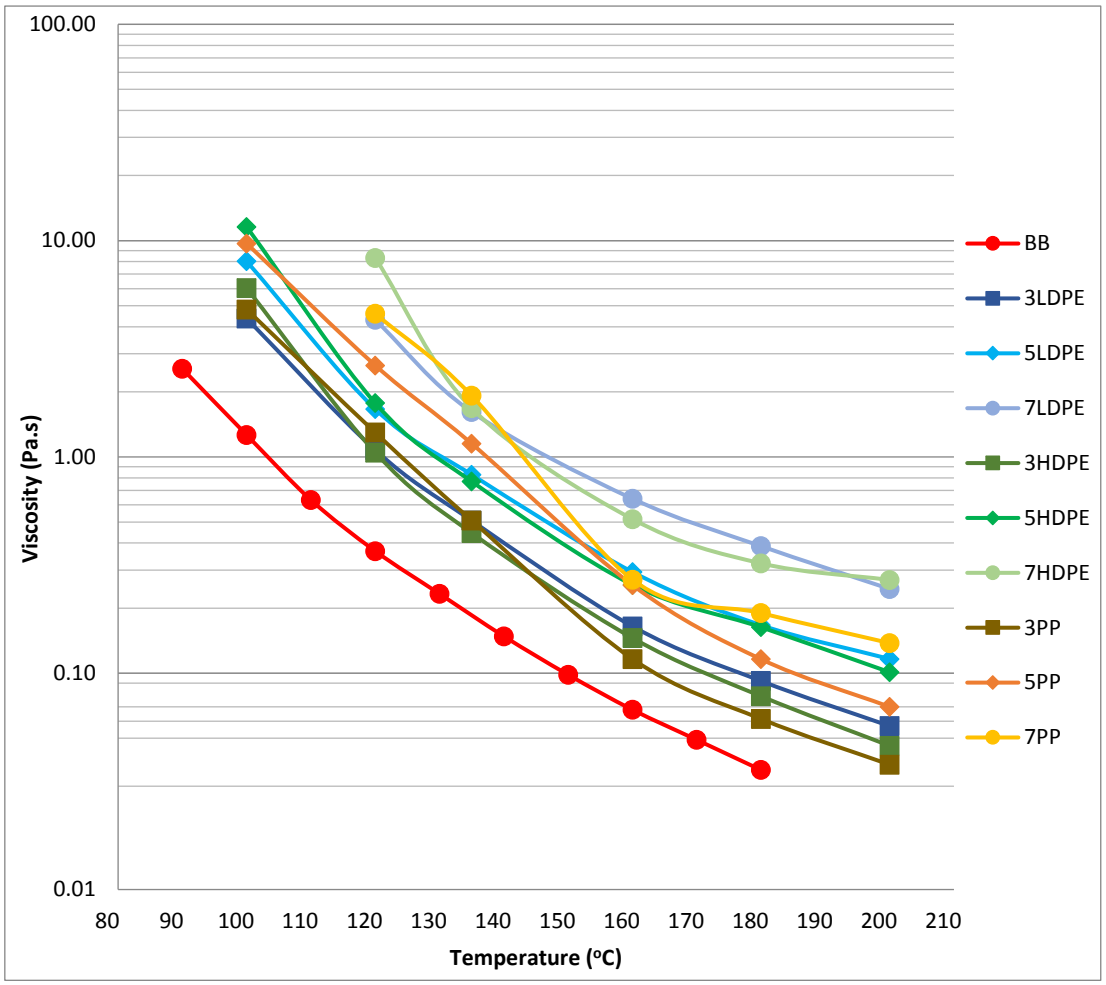


Figure 5.1: Viscosity values of RWP modified binders

5.3 Rheological Properties of RWP-MB

In order to acquire more information about the effect of different types and concentrations of RWPs used for modifying the base binder upon the rheological properties of the binder, dynamic shear rheometer (DSR) tests were performed. The basic rheological parameters which are complex modulus (G*) and phase angle (δ) were obtained from frequency sweep tests carried out in accordance with the procedures in BS EN 14770:2012 test method by using the parameters as described in Section 3.3.7.5 and the reference temperature of 25°C. Briefly, the G* can be considered as a measurement of total resistance of the binder against deformation

when repeatedly sheared and the δ is the phase lag between the applied shear stress and shear strain responses which is the indication of the balance between viscous (90^o) and elastic (0^o) deformation of the binder ((Airey 1997; Köfteci et al. 2014). The rheological characteristics of the RWP-modified binders in the linear viscoelastic range can be evaluated through master curves and Black diagrams. Asgharzadeh and Tabatabaee (2013) stated that rheological master curves provide fundamental understanding of the material because this curve allows an estimation of extended data (mechanical properties) over a wide range of temperature and frequencies (loading times) which are not tested in the laboratory but experienced in the field and also provides qualitative criteria for assessing binder performance at different temperatures. While Black diagrams could evaluate the influence of a polymer addition on the structural properties of the binder (Teugels 2000) and consists of a graph of G* versus δ in a semi-logarithmic scale (Airey 2002a; Polacco et al. 2006).

5.3.1 Master Curves

As can be seen in Figure 5.2, the shape of the master curve for RWP-modified binders are identical to the base binder especially for low and intermediate modification levels (namely 3% and 5% (except for modification of base bitumen with 5PP)) irrespective of the types of RWPs used. This means that these RWP-modified binders may have similar behaviour to the base bitumen at this level of modification. However, the complex modulus of these RWP-modified binders was higher than base bitumen and indicated that the addition of RWP into the base binder makes the binder stiffer. This happens because the modulus of the thermoplastic used is higher than the base bitumen (Da Silva et al. 2004).

The other RWP-modified binders, namely the high modification level (7% amount of RWP) and 5PP-modified binder displayed interesting behaviour. It can be seen that there is a decrease in the slope of the RWP-modified binder at low frequencies (dashed circle in Figure 5.2) producing a plateau at low reduced frequencies. This can be attributed to the molecular interaction of RWP-modified bitumen (Asgharzadeh & Tabatabaee 2013). At these equivalent low reduced frequencies – high temperatures, the RWP becomes the main contribution to the rheological behaviour of the binders and therefore improves the viscoelastic characteristics of the binder at higher temperatures (Pérez-Lepe et al. 2005). The improvement increases with RWP content but varies with the type of RWP added. The amount of RWP added produces a more significant effect on the rheological results compared to the type of RWP used as evidenced by the similar trend of RWP types observed for each level of modification.

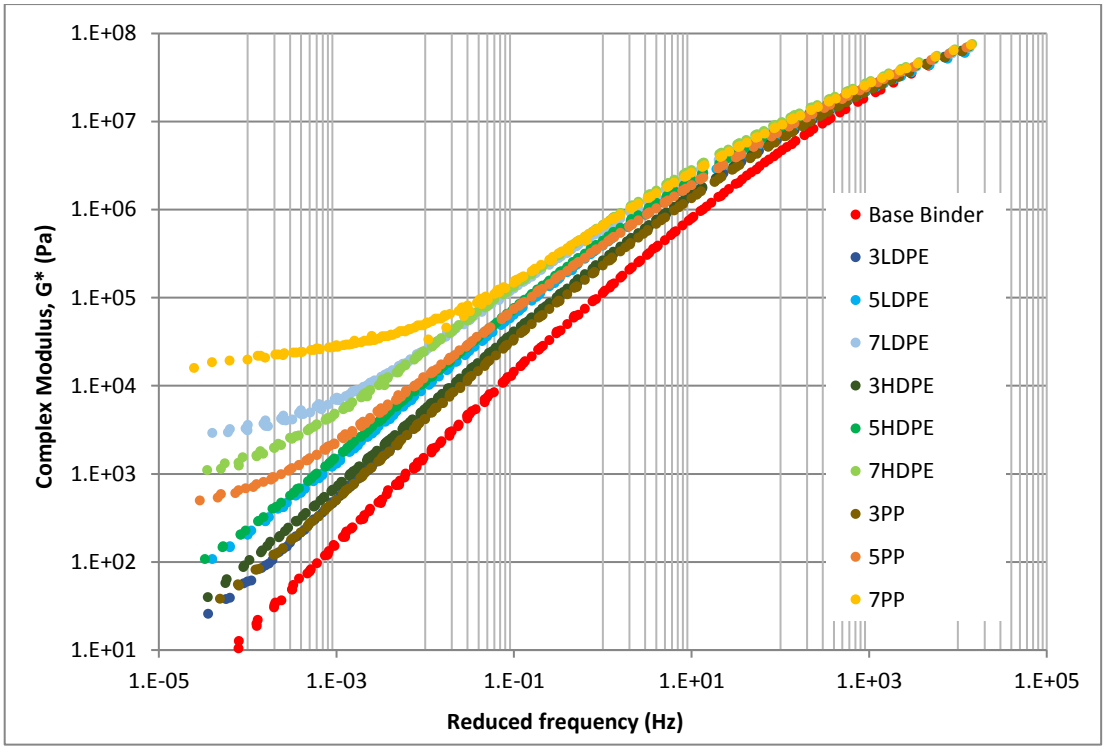


Figure 5.2: Complex modulus master curve for binders at T_f = 25°C

The master curves of phase angle for all binders at a reference temperature of 25°C are displayed in Figure 5.3. As shown, the phase angle for all modified binders exhibited a reduction over the entire frequency range compared to the base bitumen. The decrease in phase angle is more pronounced at low frequencies as the amount of RWP added increased regardless of the type of RWP. This means that the RWP-modified binders have more elastic behaviour than the base bitumen at high temperatures. At high frequencies (equivalent to low temperatures), all modified binders for each level of modification have similar phase angles, meaning that the bitumen-rich phase of the RWP-modified bitumen is dominant at this stage (Airey 1997). As the reduced frequency decreases or at the intermediate reduced frequencies range, a plateau (dashed rectangular) of the phase angle curves of RWP-modified binders appears which indicates that at this point there is a balance of

rheological behaviour influenced by both the RWP and base binder (Da Silva et al. 2004). The structural changing of the polymer itself may cause the formation of a plateau in the phase angle curve as reported by Asgharzadeh and Tabatabaee (2013). Finally, at low reduced frequencies, all the RWP-modified binders show a reduction in phase angle and increased elastic behaviour. This is due to the RWP forming a dominant polymer network and hence, the rheological properties of modified binders are dominated by the polymer network (Airey 2003). This reduction is significantly displayed when the RWP content is increased. Thus, the higher content of RWP in the base binder should have good rutting resistance as evidenced by the decreased phase angle and therefore increased elastic response.

Besides that, a unique feature can be seen in Figure 5.3 for 7PP-modified binder. It shows a break or disjoint in the curve which exists at the intermediate reduced frequencies. This may be caused by the structural change or bitumen phase transition in the binder (Asgharzadeh & Tabatabaee 2013).

From the master curves of the RWP-modified binders, it can be concluded that the advantages of adding RWP is more noticeable at low frequencies and high temperatures and the rheological properties of modified binders is strongly dependent on the amount of RWP added into the base binder rather than the type of RWP used. From this conclusion, the RWP-modified binders should be beneficial to rutting resistance at high temperatures.

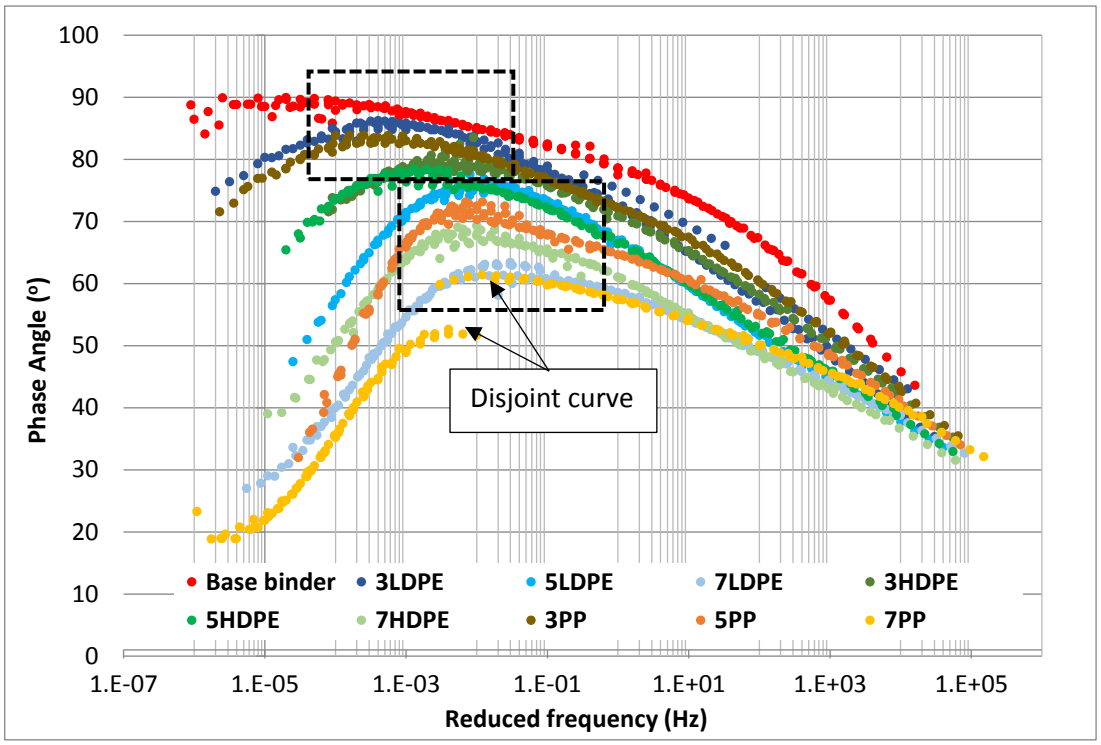


Figure 5.3: Phase angle master curve for binders at T_f = 25°C

5.3.2 Black Diagrams

The Black diagram for the unmodified and modified binders is shown in Figure 5.4. According to Figure 5.4, at different frequencies and temperatures, the Black diagram curve for each of the binders is smooth and continuous (overlapping sets of data at different frequencies and temperatures) and thus exhibits thermorheologically simple behaviour except for the 7PP-modified bitumen. It can be seen that there is a decrease in stiffness and tendency toward elastic behaviour as the temperature increases for the modified binders compared to the base bitumen which shows low complex modulus values and increased viscous response at high temperatures.

As shown in Figure 5.4, all the modified binders appear to have lower phase angles than the base bitumen. This is different to the curve of the base bitumen which exhibited highly viscous response (phase angle towards 90°) as the stiffness modulus

decreases as the temperature increased indicating that the material becomes more fluidlike (Murphy et al. 2001). However, the curve of 3LDPE-modified binder showed superimposed data over the base bitumen at phase angles between approximately 80^{o} and 88^{o}. This indicated that performance of low modification was almost similar to the base binder over that phase angle range. The curve for all RWP-modified binders tends to shift towards lower phase angles (elastic behaviour) with the value close to 20^{o} as the temperature increased which indicates that the polymer network is dominating the behaviour of the binder at high temperatures and the shift among the curves becomes more obvious when a greater amount of RWP is added irrespective of the RWP type. This means that the addition of RWP enhances the elastic properties of the binders and therefore improves their resistance to rutting at high temperatures (Pérez-Lepe et al. 2005). This improvement increases with RWP content but varies with the types of RWPs. This strongly demonstrates that the enhancement of modified binders is highly dependent on the amount of RWP added instead of types of RWP used. In term of G*, all RWP-modified binders showed similar stiffness at low temperature with the values changing significantly as the temperature increased with the decrease in G* with increase in temperature being lower as the amount of RWP increased. Besides that, the disjointed curve, as displayed in the phase angle master curves, also appears in the Black diagram for the base bitumen modified with 7% of PP and this behaviour may be related to the structure of the modified binder (Airey 1997).

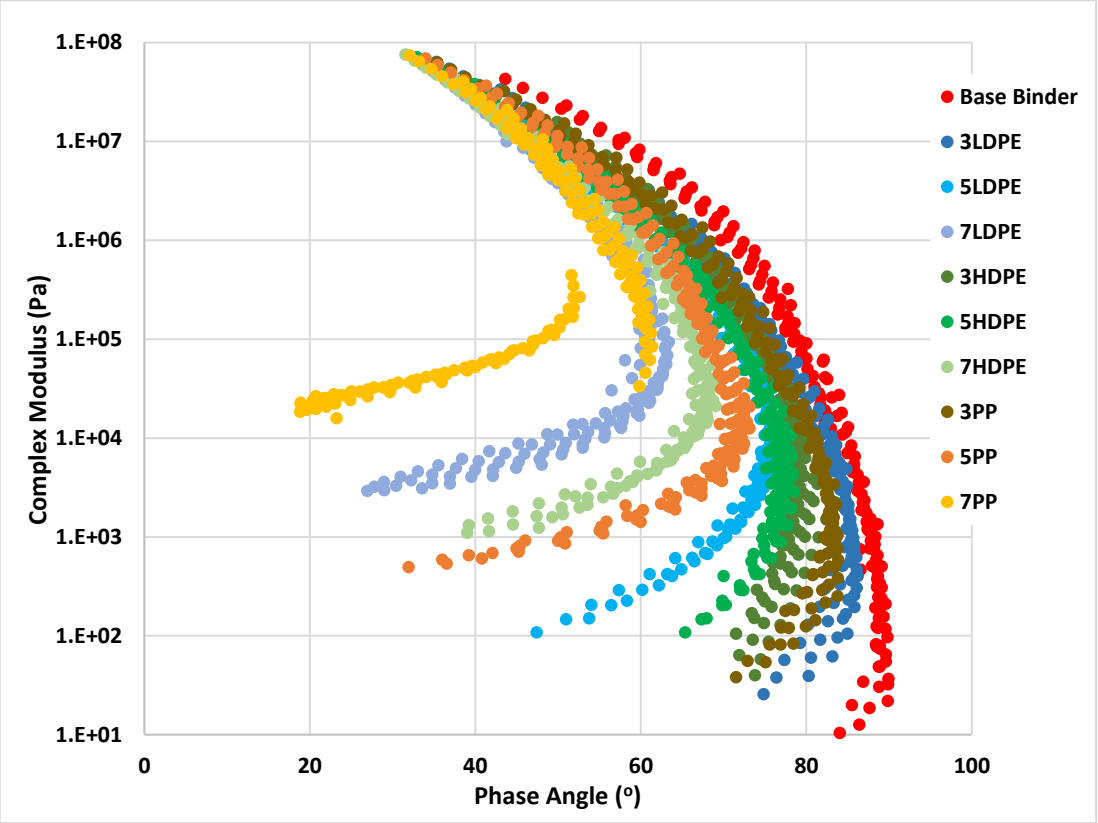


Figure 5.4: Black Diagram for unmodified and modified binders

Based on the Black diagram, it can be concluded that the changes in G* and δ caused by the RWP-modified binder results in an increase of stiffness and a decrease of phase angles (increase in elastic response) as the temperature increases and becomes more significant as the concentration of RWP increases. This highlights how the amount of RWP added and service temperature affects the properties of the binders rather than the type of RWP used. Moreover, the highly elastic component of the RWP-modified binders at high temperatures revealed that these binders would resist rutting.

5.4 Rheological Properties of Aged RWP-MB

The binders were subjected to two different ageing processes, namely short-term ageing and long-term ageing. The RTFOT was used to simulate the short term ageing process (denoted as binder_RTFOT) and then, the residue from RTFOT was used in the PAV to simulate the long term ageing process (denoted as binder_R+P as the binder experienced RTFOT and PAV ageing). The procedures of the ageing processes were described in Section 3.3.9 for RTFOT and Section 3.3.10 for PAV. The effect of ageing upon rheological properties of the bitumen were examined by using frequency sweep tests. The results of the viscoelastic properties which are complex modulus and phase angle were presented in terms of master curve at a reference temperature of 25^{o}C. Then, these viscoelastic values at 25^{o}C and 1.59 Hz were calculated to evaluate the ageing index.

The susceptibility of binders to ageing can be assessed using the ageing index in which the higher degree of binder hardening is denoted by the higher ageing index value (Rami Mohammed 2018). The ageing index can be calculated as follows:

$$Ageing\ Index\ (AI) = \frac{Physical\ property\ of\ aged\ binder}{Physical\ property\ of\ original\ binder} \qquad \text{Equation 5.1}$$

5.4.1 Master Curves

Complex modulus master curve in Figures 5.5, 5.6 and 5.7 display a stiffness evolution of binders at different stages of ageing for LDPE, HDPE and PP-modified bitumen, respectively. Analysing Figures 5.6, 5.7 and 5.8 presented similar trends hereby the different between short-term and long-term ageing is clearly observed. It shows that the complex modulus of binders increased as the binders experienced ageing

processes. The increase in stiffness is more pronounced after the RTFOT+PAV condition due to increased oxidation and loss of the light components of bitumen materials making the binder harder. This is because the binders have undergone severe ageing namely short-term ageing with RTFOT and then long-term ageing by PAV.

Examining Figures 5.5, 5.6 and 5.7 at higher frequencies (fast moving vehicle or high speed road) corresponding to low temperatures exhibited the complex modulus values converging towards a common threshold value regardless of binder types and ageing conditions. While at low frequencies (slow moving vehicle or low speed road) and higher temperatures, the discrepancy of complex modulus among the binders at different ageing conditions are significant with the RTFOT+PAV ageing binders have the highest complex modulus values followed by the binder of RTFOT ageing and then unaged binders. Moreover, it can be noticed that the slope of all RWP-modified bitumen for the binders that experienced RTFOT+PAV ageing condition decreases at low frequencies due to the presence of RWP in the binder in which the polymer network is dominant. The higher complex modulus of RWP-modified bitumen should be beneficial for the high-temperature permanent deformation resistance and probably low-temperature cracking (Liu et al. 2013).

In order to make the verification of visual analysis shown on the master curves quantitatively, ageing indices (AI) were calculated using the data of complex modulus values at temperatures of 25^{o}C and 65^{o}C and loading frequency of 1.59Hz and displayed in bar charts. The complex modulus values at temperature of 25^{o}C and 65^{o}C and loading frequency of 1.59 Hz are presented in Figure 5.8 and Figure 5.9,

respectively. Regarding the change in complex modulus ageing index at temperature of 25^{o}C, it shows a decreasing trend with increasing amounts of RWP. After long-term ageing, the trend is not as clear with higher ageing indices for two of the 7% modified binders (except for 7LDPE-modified bitumen). The ageing index for the modified binders is still below that of the base bitumen. This indicates that the base bitumen has higher degree of bitumen hardening compared to modified binders when considering the rheological data at 25^{o}C. Using rheological data at 65^{o}C, no clear trend could be observed but at the RTFOT ageing stage, the higher modification level exhibited lower values of ageing index compared to the base bitumen. However, after further ageing with the PAV the ageing index was greater for the modified binders especially for 7HDPE and 7LDPE-modified bitumens. A decrease and increase of ageing index at temperatures of 25^{o}C and 65^{o}C respectively will make the aged RWP-modified bitumen better in terms of resistance to rutting and low temperature cracking (Airey 1997).

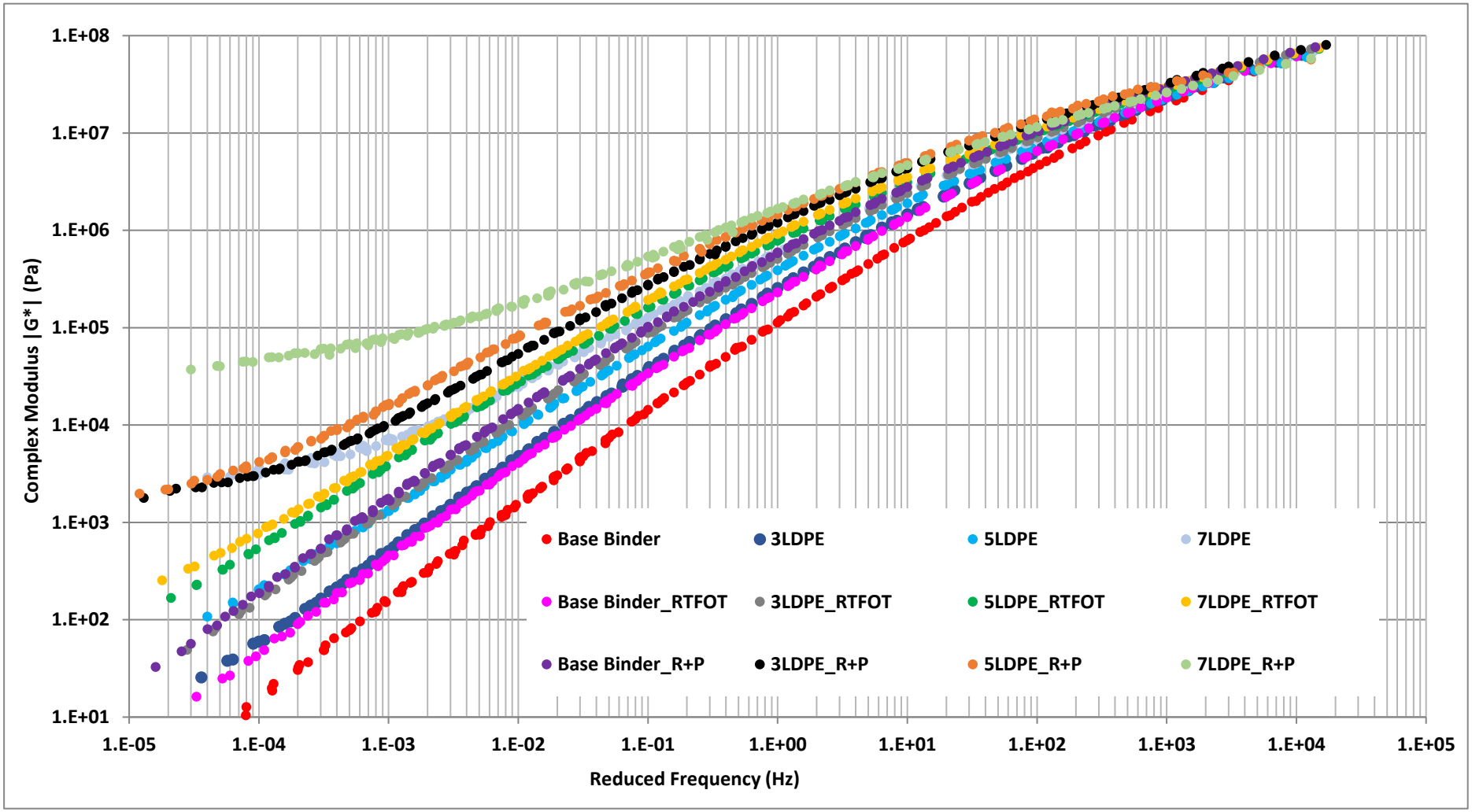


Figure 5.5: Complex modulus master curve for base bitumen and LDPE modified bitumen at T_f = 25°C

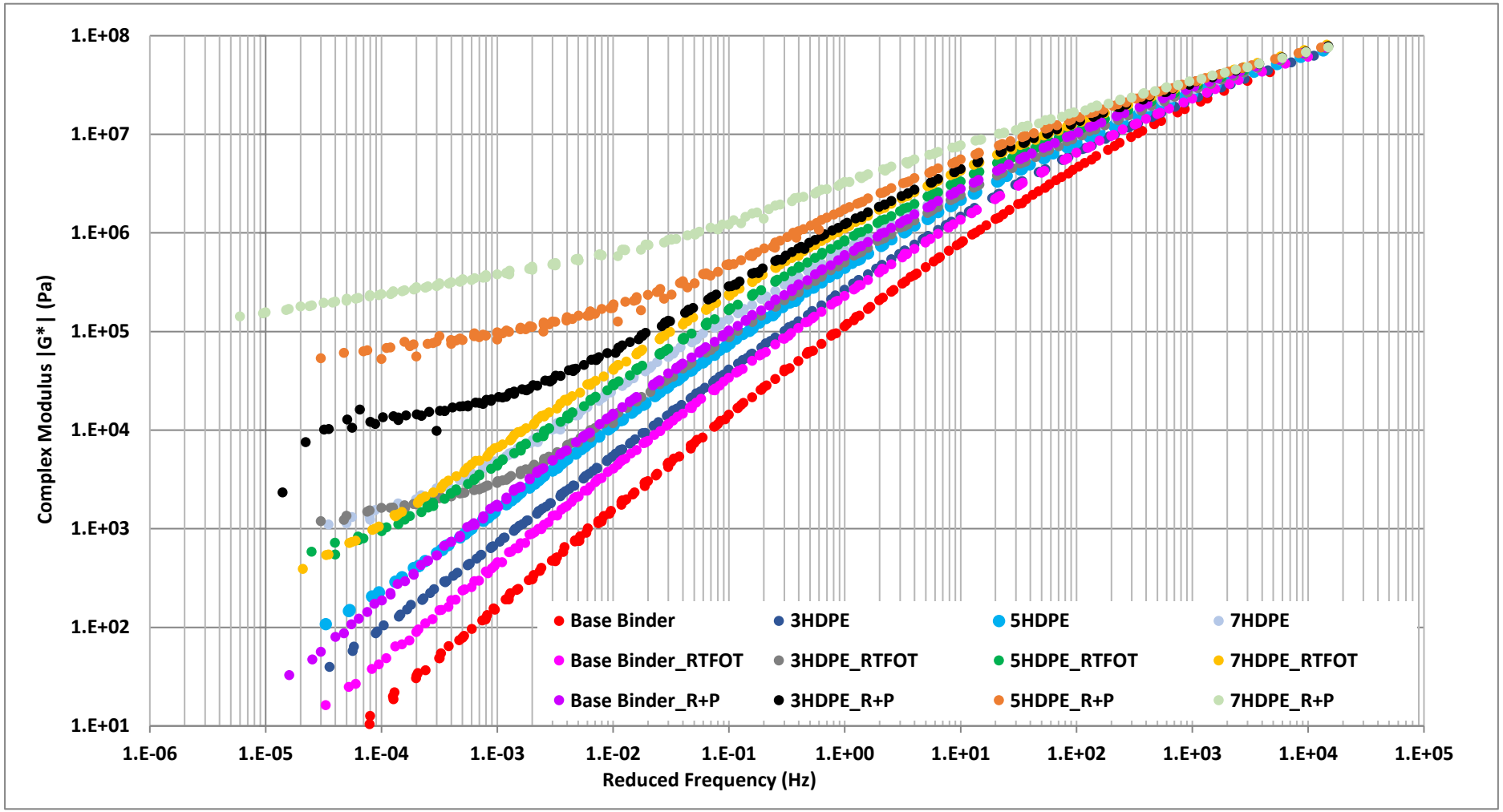


Figure 5.6: Complex modulus master curve for base bitumen and HDPE modified bitumen at T_f = 25°C

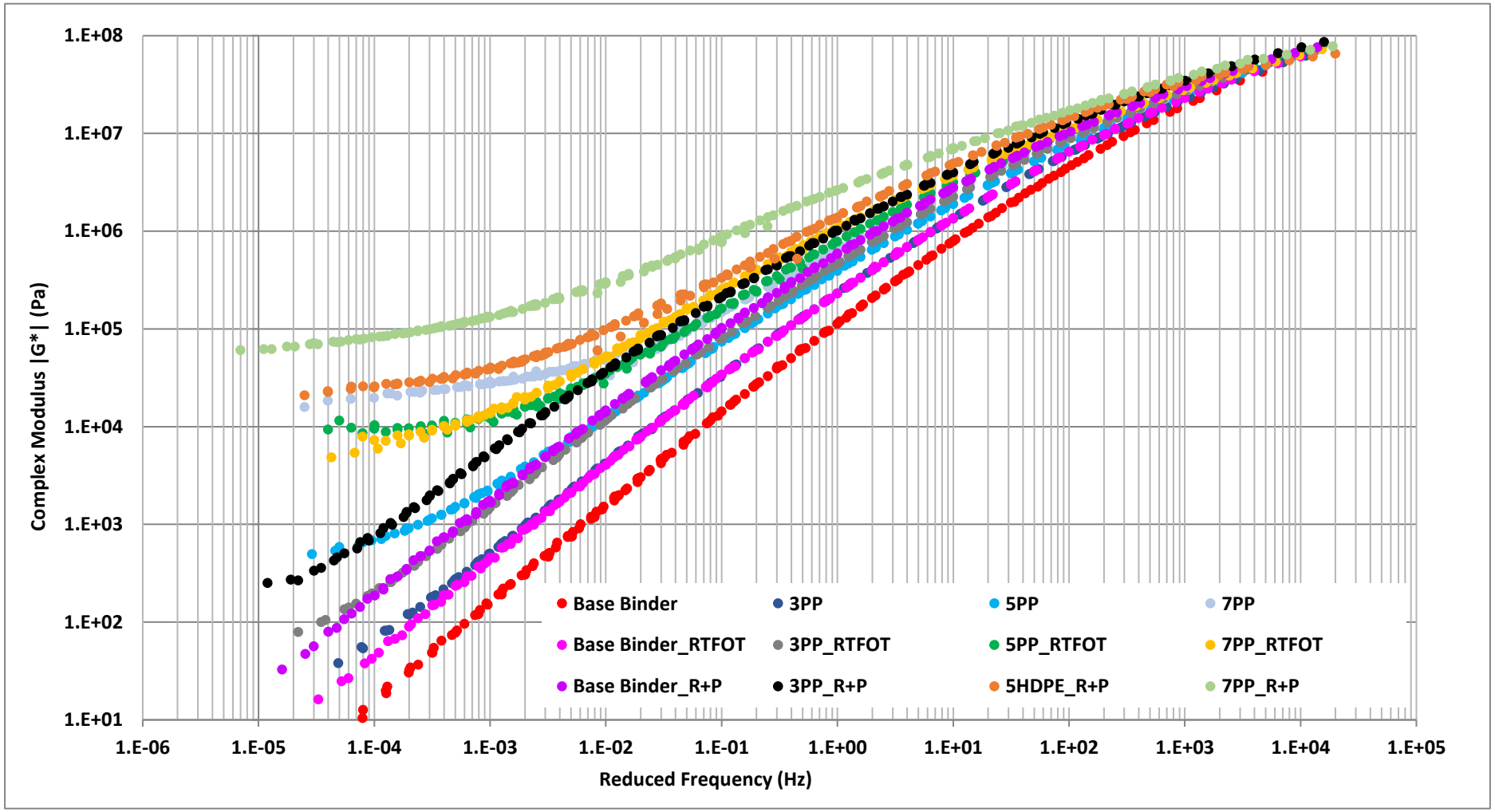


Figure 5.7: Complex modulus master curve for base bitumen and PP modified bitumen at T_f = 25°C

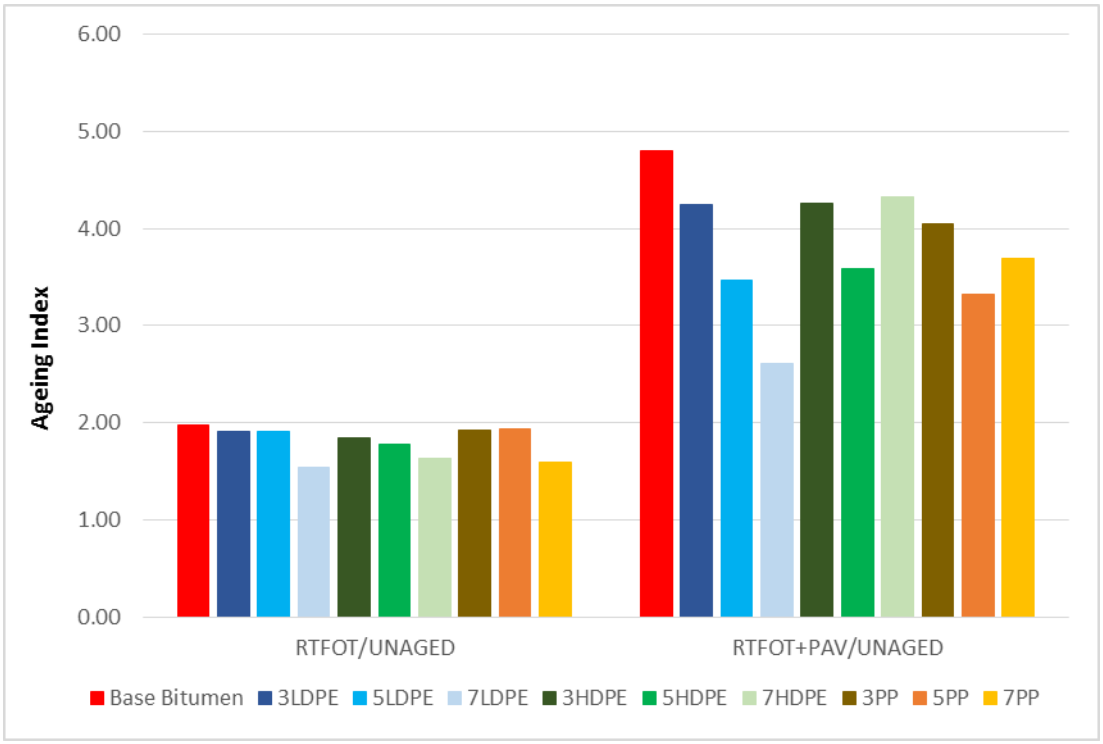


Figure 5.8: Complex modulus ageing index of modified bitumen (G* at 25°C and 1.59 Hz)

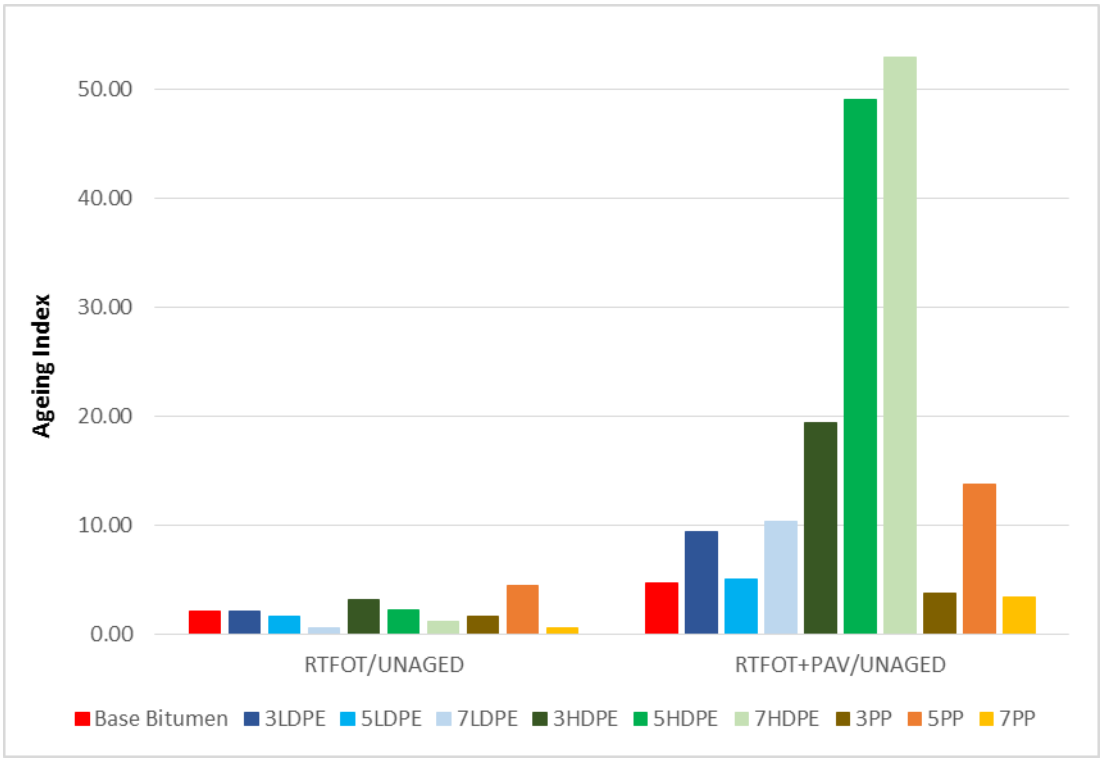


Figure 5.9: Complex modulus ageing index of modified bitumen (G* at 65°C and 1.59 Hz)

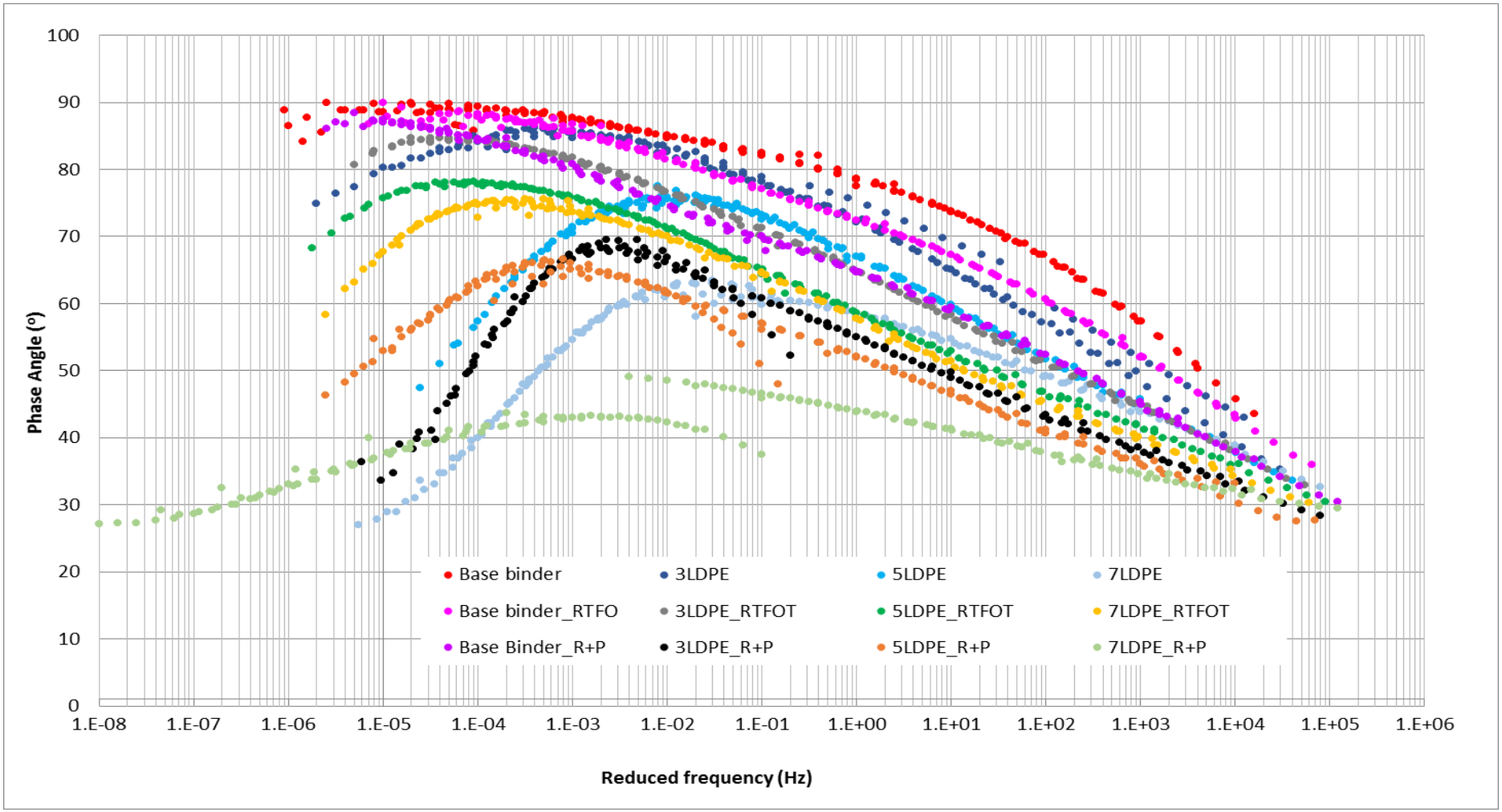


Figure 5.10: Phase angle master curve for base bitumen and LDPE modified bitumen

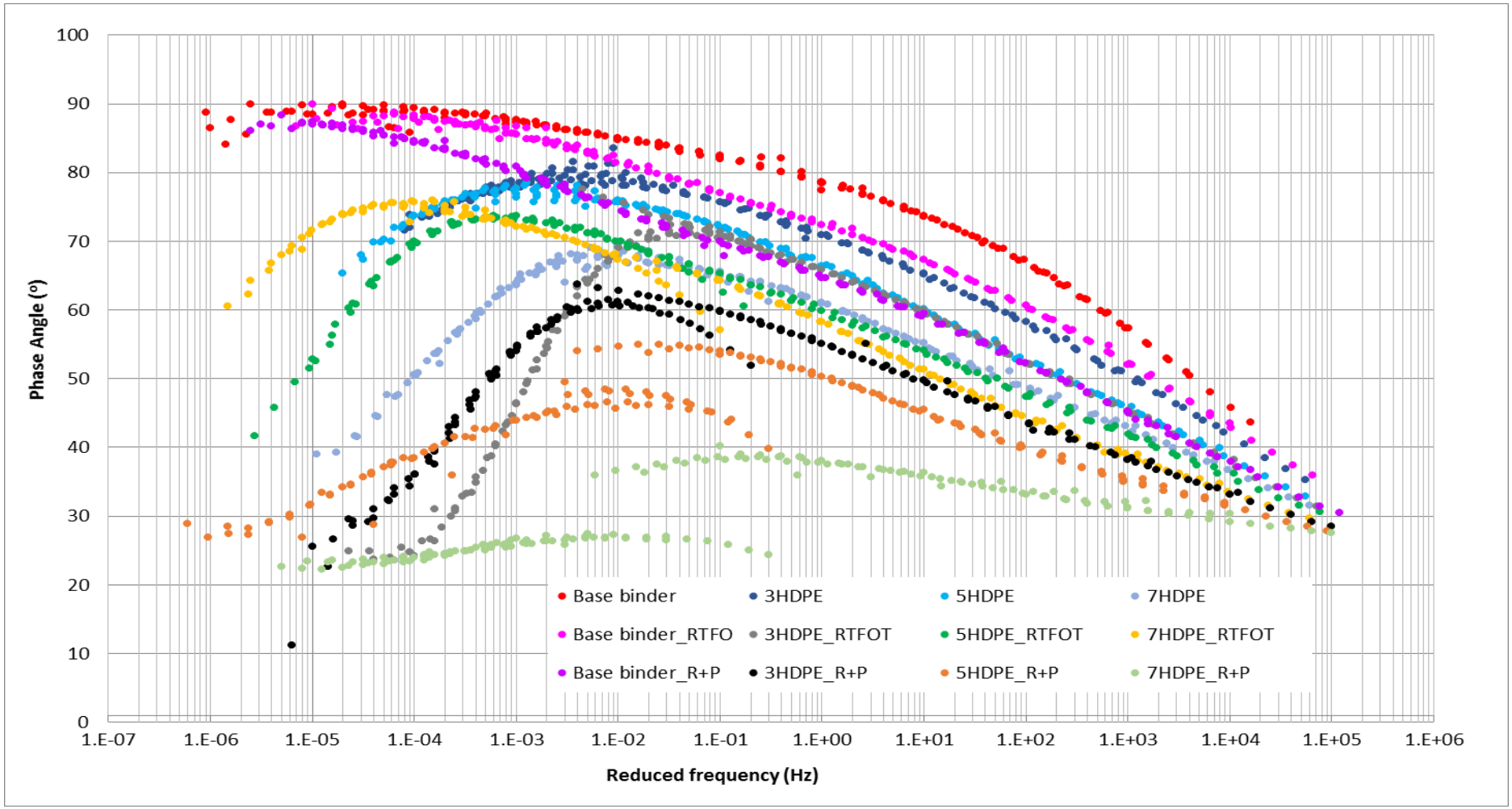


Figure 5.11: Phase angle master curve for base bitumen and HDPE modified bitumen

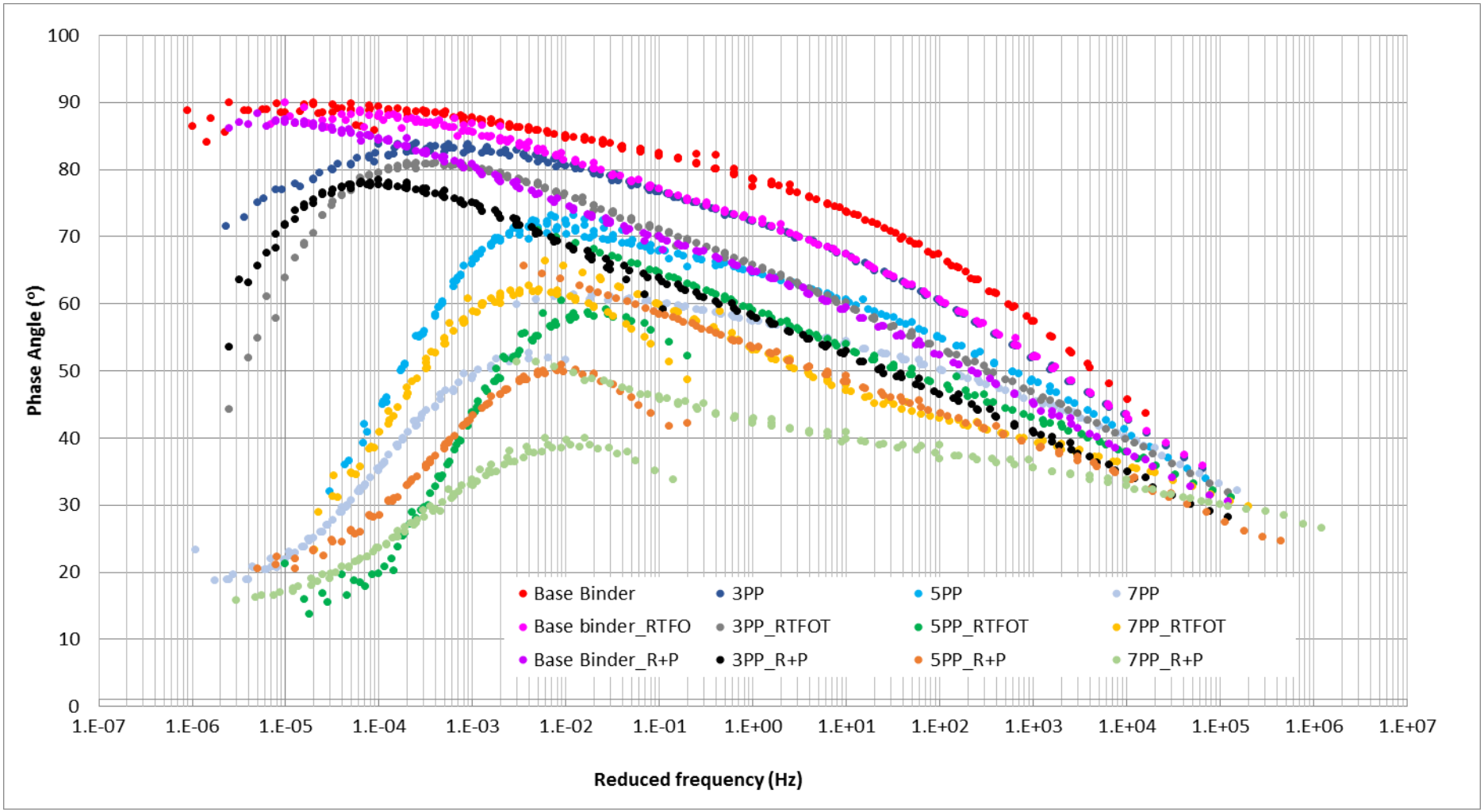


Figure 5.12: Phase angle master curve for base bitumen and PP modified bitumen

Figures 5.10, 5.11 and 5.12 present the changes of phase angle with frequency at different ageing conditions for LDPE, HDPE and PP-modified bitumen, respectively. The phase angle of RWP-modified bitumen was lower than that of the base bitumen over the entire frequency range. At higher frequencies, corresponding to low temperatures, the phase angle of all the binders demonstrated a similar trend as shown for the complex modulus master curves with only a slight difference in phase angle for different RWP-modified bitumens and this difference almost disappeared as the curves converged towards phase angle values around 30^{o}.

While at intermediate frequencies range, a plateau region is observed in the phase angle master curve for all RWP-modified bitumens at all ageing conditions which indicates that the compatibility between the base binder and the RWP is better even after undergoing several ageing conditions. However, the addition of 5% of HDPE and PP and 7% for all type of RWPs display a break on the phase angle curve particularly at the plateau region after RTFOT+PAV ageing method. Moreover, the formation of this break is almost at similar reduced frequency for all type of RWPs which may cause by the compliance errors related to the testing apparatus.

Then, at low frequencies range, the different in phase angle for each ageing stage is pronounced. The short-term ageing (RTFOT) increases the elastic behaviour (decrease in phase angle value) of the RWP-modified bitumen. The reduction of phase angle is more significant with further ageing and more noticeable when different amounts of RWP are added. The phase angle curve tends to become a slightly flat particularly for the 5 and 7% of RWP-modified bitumens regardless of

RWP used which might be due to the composition changes of the modified binder (Zhang et al. 2010).

Individual phase angle values for the RWP-modified bitumen before and after RTFOT and RTFOT+PAV are presented in Table 5.3. As seen in Table 5.3, all the binders have a similar trend for both temperatures with a movement towards a more elastic behaviour after ageing. The increase in elasticity of the binders is significant with the RWP and more pronounced when the amount of RWP addition is greater after further ageing. However, at high temperature, the binders of 7% modification level have more viscous response as evidenced by higher phase angles but the phase angles then decrease (increased elastic response) after RTFOT+PAV ageing. Overall, it can be seen that the complex modulus increases and the phase angles decrease with ageing and thus the binders behave more solid-like (Lu & Isacsson 2002).

Table 5.3: Phase angle of RWP-modified bitumen at loading frequency of 1.59 Hz for two different temperatures

Bitumen		δ at 25°C			δ at 65°C		
		Unaged	RTFOT	RTFOT+PAV	Unaged	RTFOT	RTFOT+PAV
Base Bitumen		77.74	71.50	63.65	88.89	86.89	85.03
LDPE	**3**	71.15	63.39	53.79	85.27	83.96	62.25
	5	65.57	57.24	50.81	73.54	77.93	64.26
	7	57.76	56.31	43.28	49.03	75.36	34.85
HDPE	**3**	69.78	64.67	53.94	79.24	48.02	43.28
	5	65.10	58.58	49.35	77.38	71.36	38.50
	7	59.75	56.77	37.55	63.25	75.50	24.38
PP	**3**	71.31	64.57	56.90	82.58	80.55	77.69
	5	63.92	57.86	52.47	67.36	36.04	34.21
	7	56.72	51.80	41.70	28.89	50.29	26.01

5.5 Fatigue

Fatigue cracking is one of the main distresses of a pavement structure that occurs at intermediate temperatures between 10 to 40^{o}C. This distress is caused by repeated loading whereby the crack will appear and degrade the bound pavement materials and then propagate into the pavement structure (Cocurullo et al. 2008). The effect of adding RWP into base binder on fatigue was evaluated by means of the Linear Amplitude Sweep (LAS) test.

5.5.1 Linear Amplitude Sweep (LAS) Test

According to AASHTO, the LAS test is performed to evaluate the ability of a binder to resist damage by means of cyclic loading at increasing amplitudes in order to accelerate damage (AASHTO, TP101-1). The test is conducted as described in Section 3.4.1.1 at three temperatures namely 15, 20 and 25^{o}C by using the DSR with parallel plates of 8 mm diameter and 2 mm gap. All binders were subjected to the RTFOT and PAV procedures to stimulate the long-term ageing effect as fatigue cracking usually occurs after years of service life of pavements (Jiménez del Barco Carrión 2017).

5.5.1.1 Failure Strain Comparison of Binders

According to the curve of shear stress as a function of shear strain, the time of damage occurring during the test is represented by a significant reduction in shear stress after being subjected to the constant strain amplitudes. The binders would not experience any damage if the shear stress response is increase when the strain applied increased (Johnson 2010; Jafari & Babazadeh 2016). Masad et al. (2001); Xue et al. (2017) stated that the failure point of the binders on the LAS stress-strain curve

is identified based on the peak stress and this point is known as a yield threshold of the binder when subjected to the increasing load.

The plot of shear stresses versus shear strains for each binder tested at temperatures of 15, 20 and 25^{o}C obtained from LAS test are shown in Figures 5.13, 5.14 and 5.15, respectively. All figures showed parallel trends namely as the level of modification increased, the peak stress is significantly increased with the increasing shear strain.

By referring to Figure 5.15 at a tested temperature of 25^{o}C, the shear stress response of base binder and low binder modification (3%) show a significant degradation at strain levels of 10 to 15% with the base binder displays the lowest yield stress, followed by the 3% modification binders regardless of RWP types. Moreover, the binders containing 5 and 7% of RWP (except for 7LDPE and 7HDPE-modified binder) exhibited similar decrease in stress response at the strain level between 15 to 18% but different in the peak stress. The exceptional binders, namely 7LDPE and 7HDPE-modifed binders, demonstrated a different trend of degradation, where the stress response of these binders tends to decrease insignificantly at the end of the applied strain amplitude during the test, indicated that these binders have the least damage as evidenced by the insignificant decrease in shear stress response at higher level of strain. It is worth mentioning that the 7% modification binders may able to be subjected to strain amplitude more than 30% because these binders did not exhibit significant degradation in stress response at 30% strain. However, the degradation of shear stress response becomes pronounced when the test temperature decreased.

A similar comparison can be made according to the degradation of the shear stress for test temperatures of 15 and 20^{o}C in Figures 5.13 and 5.14, respectively. The temperature has a major influence on the fatigue resistance of all tested binder in which the increase in temperature make the damage occurred sooner. Moreover, as the test temperature decreases, the yield threshold value of base binder becomes almost similar with the shear stress degradation of 3% RWP-modified binder as shown in Figure 5.13. It is possible for this peak value of base binder to become similar or higher than the RWP-modified binder if tested at a temperature lower than 15^{o}C due to the lower stiffness for the base binder meanwhile the stiffness of the RWP-modified binder is higher at temperature lower than 15^{o}C. This means that the RWP-modified binder may have good fatigue resistance at pavement temperature more than 15^{o}C and the resistant to fatigue is increase as the modification level increased. Hence, it can be concluded that the effect of fatigue resistance on modification of base binder with RWP is mainly dependent on the amount of RWP added.

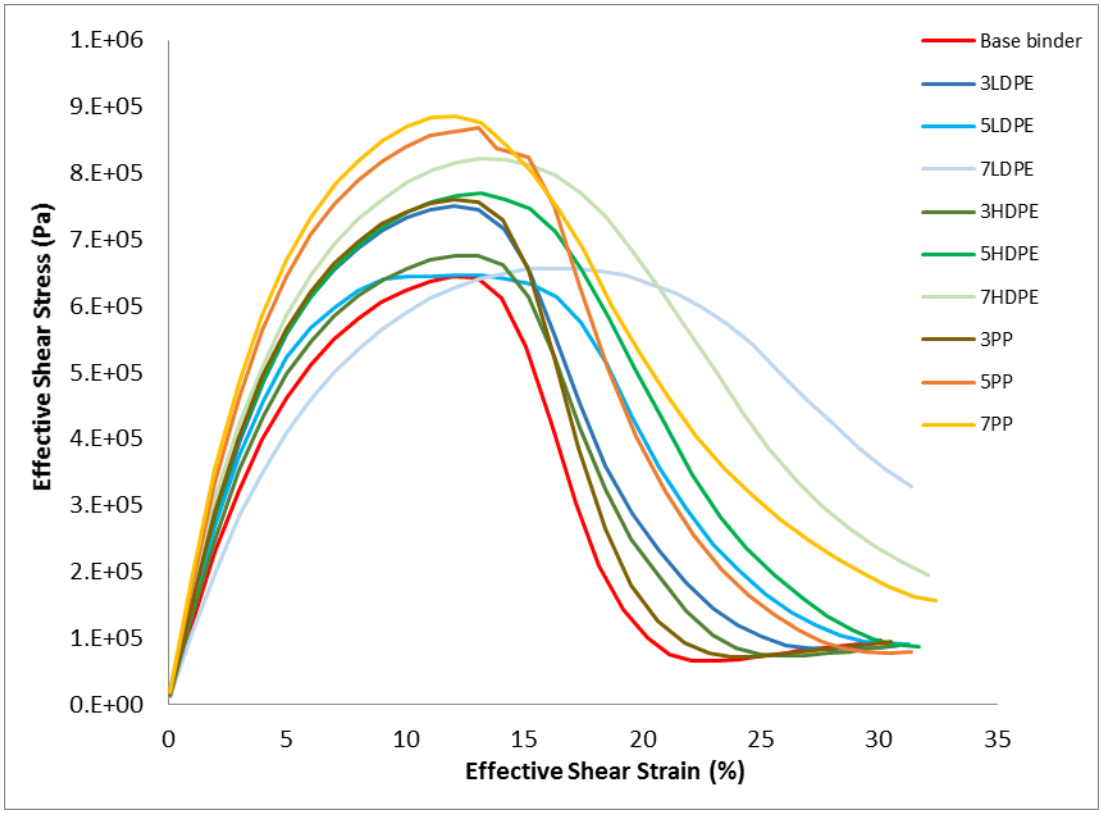


Figure 5.13: Stress versus strain curve from the LAS test at temperature of 15°C

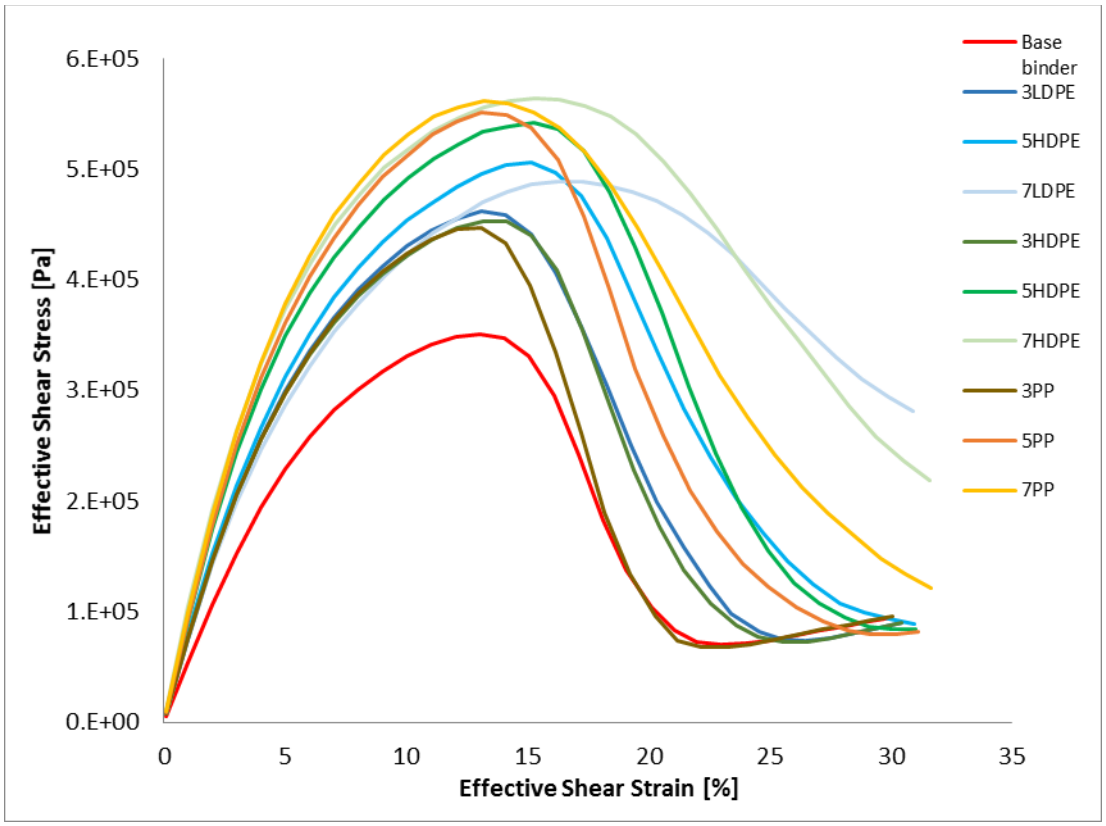


Figure 5.14: Stress versus strain curve from the LAS test at temperature of 20°C

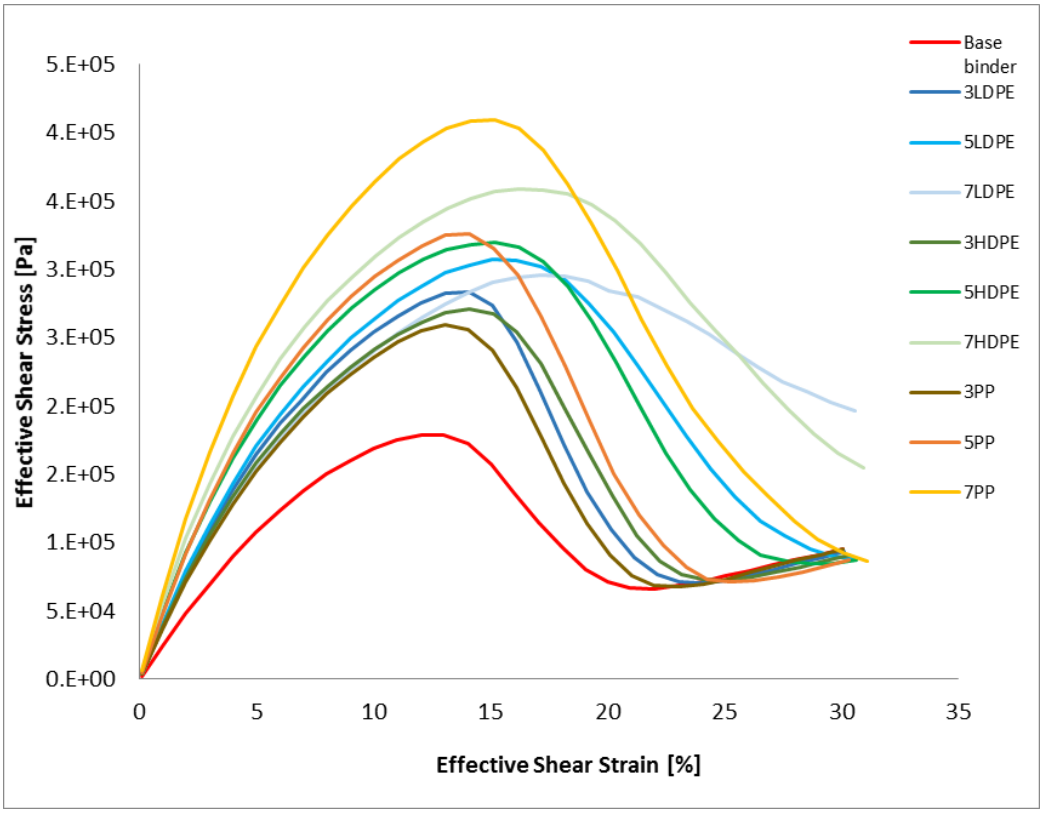


Figure 5.15: Stress versus strain curve from the LAS test at temperature of 25°C

5.5.1.2 Damage and Failure Analysis

The capability of a binder to resist damage can be interpreted from the relationship in which the binder has less reduction of $|G^*|(t)$ from its initial $|G^*|$ value indicating that this binder has lower strain susceptibility and in other words is more resistant to damage (Jafari & Babazadeh 2016; Jafari et al. 2017). Unfortunately, Xue et al. (2017) stated that this relationship is only able to evaluate the damage evolution and the fundamental material property for the fatigue life prediction. As mentioned earlier in LAS analysis procedure, the material integrity parameter $C(t)$ is the ratio of $|G^*|(t)$ to $|G^*|_{initial}$. Thus, based on this ratio, it revealed that the stiffness of binders strongly affects the position of the damage characteristic curve wherein the soft and stiff binders tend to have a lower and higher position of damage characteristic curve, respectively (Xue et al. 2017).

Figures 5.16, 5.17 and 5.18 shows the damage characteristic curves plotted between material integrity (C) as a function of damage intensity for all binders at different tested temperature namely 15^{o}C, 20^{o}C and 25^{o}C, respectively. The trend of reduction in initial $|G^*|$ value for each temperature can be seen clearly in Figures 5.16 to 5.18. It showed that at all temperatures, the resistance to damage increased as the modification level increased regardless of the types of RWP used with the most vulnerable to damage being reported for the base bitumen and the modification with low percent of RWP namely 3%. The 3% RWP modified binders tend to produce parallel behaviour with that of the base binder in which the reduction in $|G^*|(t)$ value versus its $|G^*|_{initial}$ value is less for RWP-modified binders and thus, more resistant to damage. However, by referring to the small picture in each figure, the 7% RWP-modified bitumen showed similar trends of damage characteristics curves for all tested temperatures wherein the 7LDPE-modified bitumen has the highest resistance to damage followed by 7HDPE and 7PP-modified bitumen. Therefore, higher modification is more favourable for increasing the damage resistance.

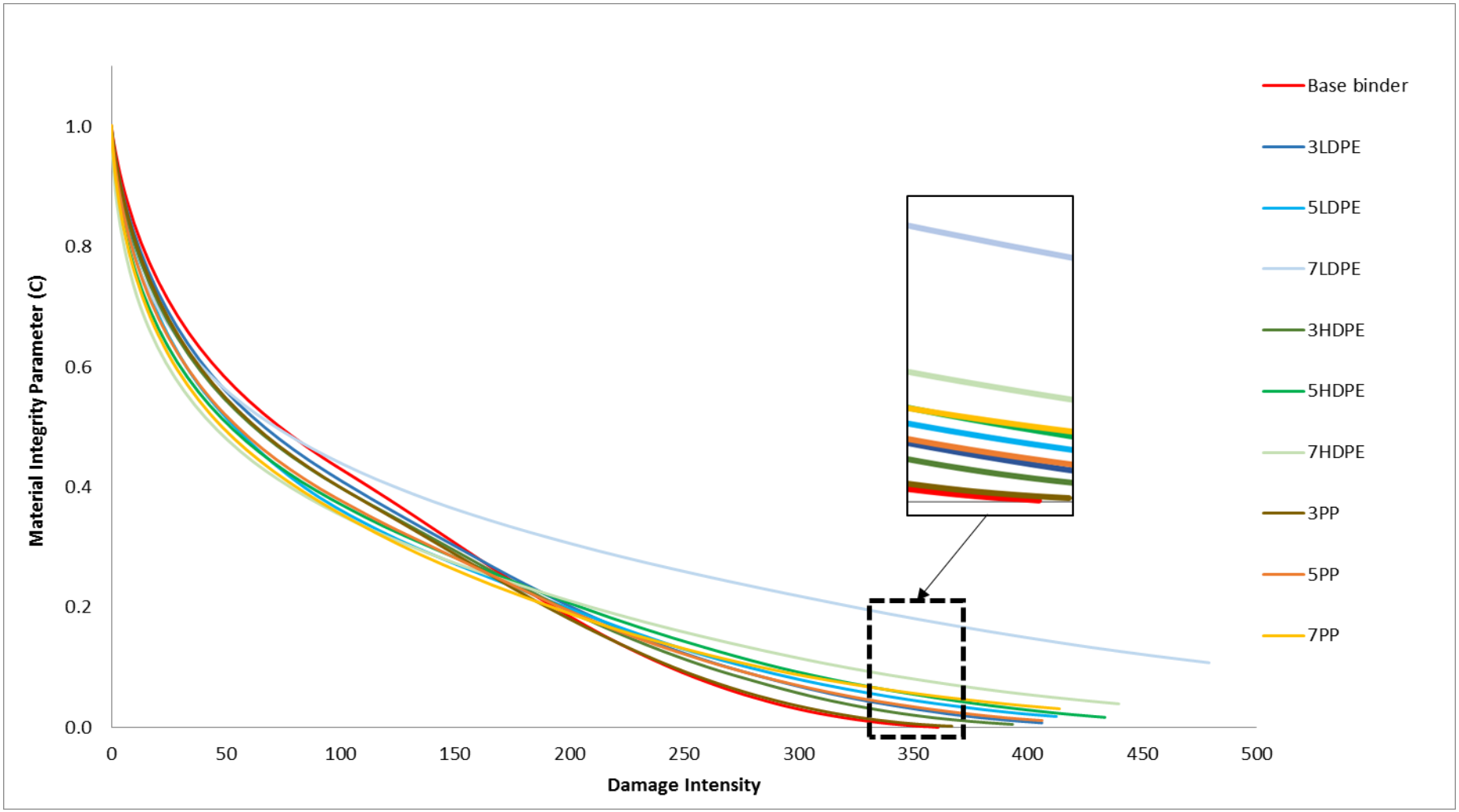


Figure 5.16: Damage characteristic curves for binders at temperature of 15°C

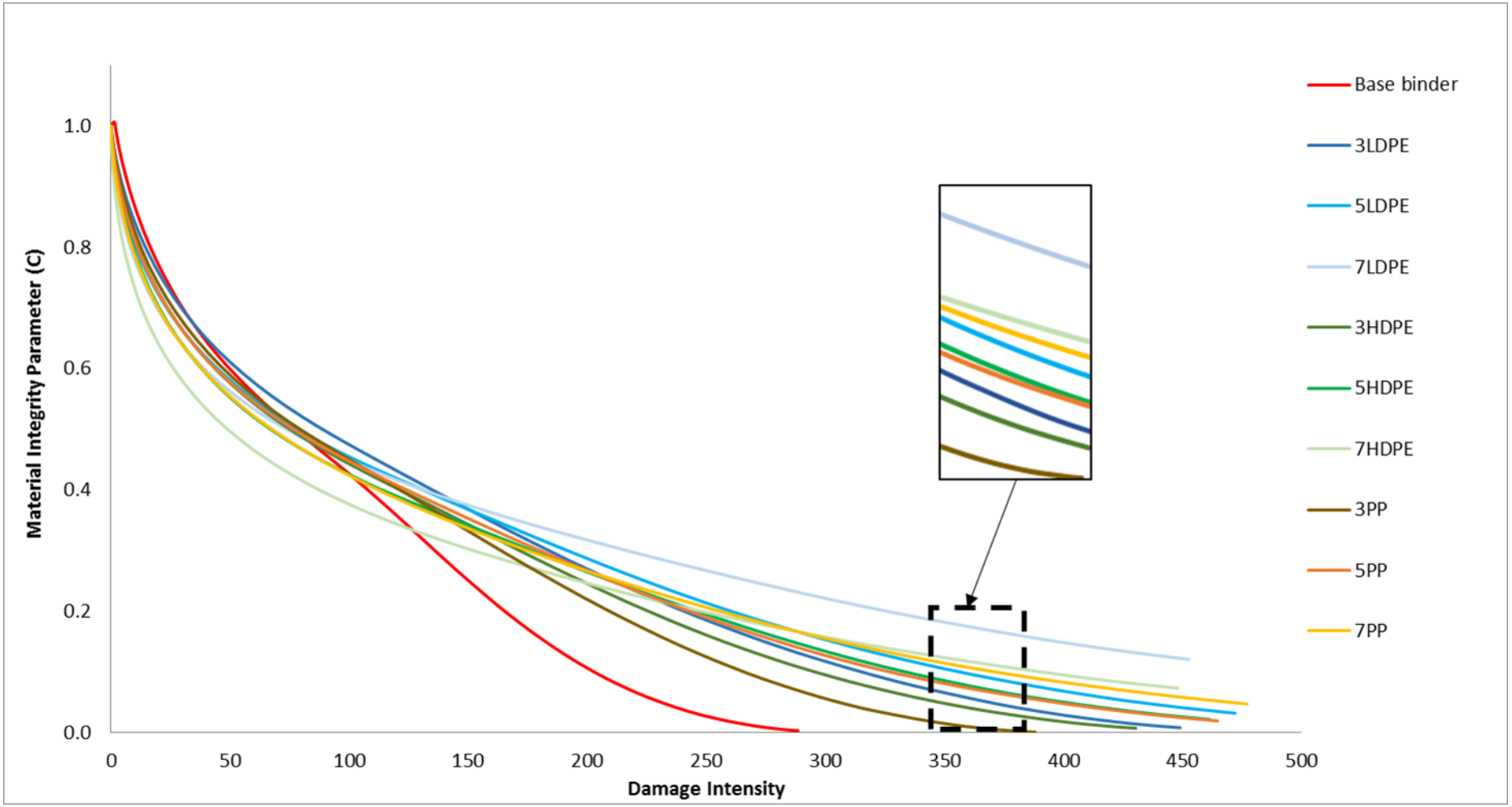


Figure 5.17: Damage characteristic curves for binders at temperature of 20°C

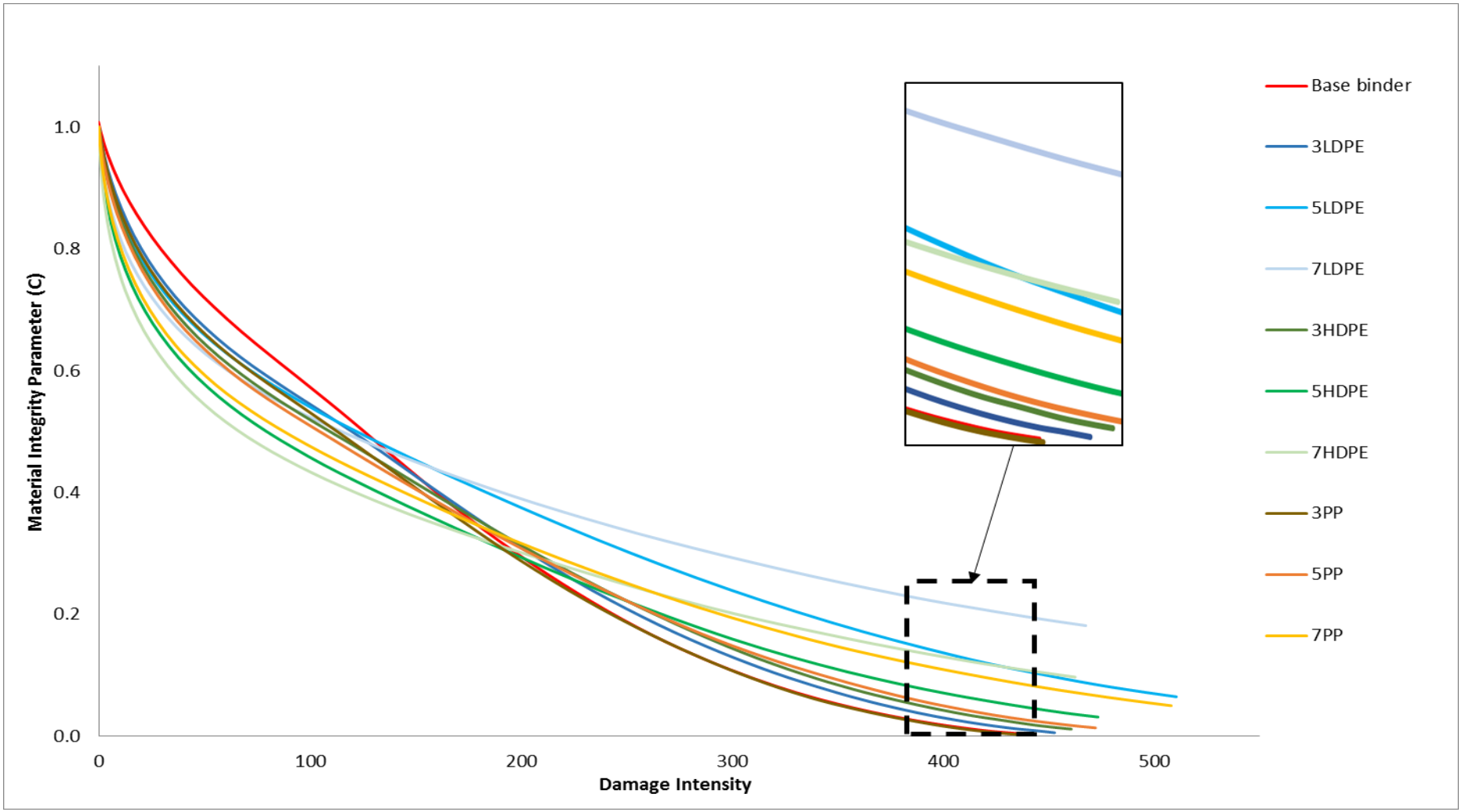
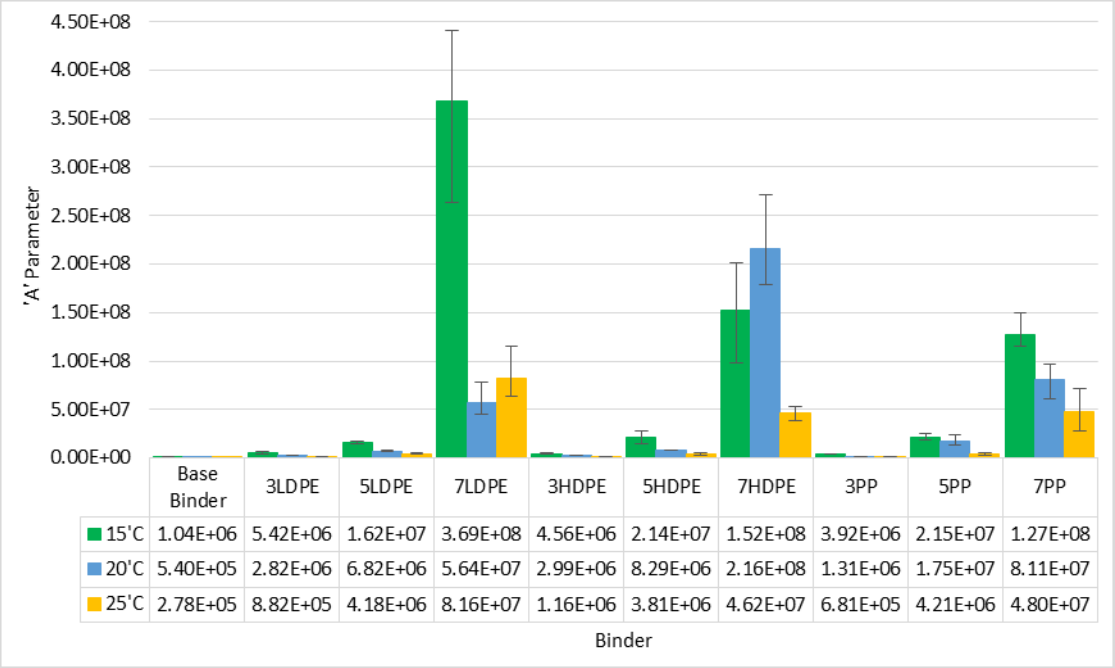


Figure 5.18: Damage characteristic curves for binders at temperature of 25°C

5.5.1.3 Coefficients of Fatigue Characteristics



	Base Binder	3LDPE	5LDPE	7LDPE	3HDPE	5HDPE	7HDPE	3PP	5PP	7PP
15'C	1.04E+06	5.42E+06	1.62E+07	3.69E+08	4.56E+06	2.14E+07	1.52E+08	3.92E+06	2.15E+07	1.27E+08
20'C	5.40E+05	2.82E+06	6.82E+06	5.64E+07	2.99E+06	8.29E+06	2.16E+08	1.31E+06	1.75E+07	8.11E+07
25'C	2.78E+05	8.82E+05	4.18E+06	8.16E+07	1.16E+06	3.81E+06	4.62E+07	6.81E+05	4.21E+06	4.80E+07

Figure 5.19: LAS test fatigue 'A' parameter at testing temperature of 15°C 20°C and 25°C



Figure 5.20: LAS test fatigue 'B' parameter at testing temperature of 15°C 20°C and 25°C

Another damage coefficient that can be identify from LAS test are parameters ‘A’ and ‘B’. The ability of the material to keep its integrity during loading or in other words to resist damage is represented by the parameter ‘A’ while the parameter ‘B’ refers to the sensitivity of the binder to the change in strain level (Moghadas Nejad et al. 2017). According to the fatigue law, the plot of the fatigue life versus strain level in log-log scale would yield the parameter ‘A’ and ‘B’ namely the intercept of the curve and the slope of the fatigue line, respectively. Generally, higher values of parameter ‘A’ indicate that the number of cracks formed in the pavement is low and hence, higher resistance to damage. Meanwhile, a lower absolute value of parameter ‘B’ indicates that the fatigue life decreases at a lower rate when strain amplitude increases or in other word, lower susceptibility to the strain level. In fact, binder having a higher ‘A’ value and lower absolute ‘B’ value is believed to have better fatigue resistance. Figures 5.21 and 5.22 provide graphs of fatigue law parameter ‘A’ and parameter ‘B’, respectively, for base binder and RWP-modified binders at three different test temperatures namely 15^{o}C, 20^{o}C and 25^{o}C with the error bars denoting the maximum and minimum value of replicates.

Incorporating the RWP into the base binder effectively increases the parameter ‘A’ value as shown in Figure 5.19 and the increase is more significant when the addition of RWP is increased. Hintz (2012) stated that the fatigue law parameter ‘A’ is temperature dependent and the parameter should exhibit increased values as the temperature increases in order to give the positive effect on the fatigue performance. However, in this study, the parameter ‘A’ value decreased when the tested

temperature is increased particularly for 3 and 5% RWP-modified binders regardless of RWP type while for 7% modification level, no specific trends could be identified.

As can be observed in Figure 5.20, the absolute value of parameter 'B' is lowest for the base binder but increases for the binder modified by the RWP with higher values being found as the amount of RWP addition is increased. Conversely, in order to have a better effect on the fatigue life, the binder should have lower parameter 'B' absolute value because higher values of this parameter indicate that the slope of the fatigue line is greater or in other words, the fatigue life decreases at a higher rate (Moghadas Nejad et al. 2017; Sabouri et al. 2018). Thus, the binders that have a higher absolute value of parameter 'B' are more susceptible to the change in strain level. Besides that, the modified binders have about the same value of 'B' parameter for each level of modification. In term of testing at different temperatures, the absolute value of parameter 'B' decreases gradually as the temperature increases. This means that all binders have lower susceptibility to the strain level.

All these results show that incorporating RWP into base binder results in higher values of parameter 'A' and the absolute value of parameter 'B'. This indicates that the binder modified by the RWP has higher resistance upon fatigue cracking during loading but is more susceptible to the change of strain amplitudes, thus the rate decrease in fatigue life is higher. Hence, the fatigue live prediction from the plot of the number of cycles to failure against strain amplitude is used to verify these results and is discussed in the next section.

5.5.1.4 Fatigue Lives Prediction of Binders

The prediction of the fatigue life is determined from the plot of the number of cycles to failure known as fatigue life against the applied shear strain. Figures 5.21, 5.22 and 5.23 show the comparison of fatigue life for the base binder and all modified binders at three temperature considered in the study namely 15^{o}C, 20^{o}C and 25^{o}C, respectively. The strain level of 1 to 10% is used to display the fatigue life in all figures because Masad et al. (2001) reported that the strain level experienced by the asphalt mixture is about 90 times of the strain level of the its binder and thus, strain level more than 10% may never occur on the road pavement.

As shown at all tested temperatures, the fatigue performance of the base binder significantly increases as the RWP is added and become better in fatigue resistance as the RWP content increases. This indicates that the high modification levels give better fatigue lives. Unfortunately, the fatigue line of RWP-modified binders is decreased steeply compared to the base binder as the applied strain level increased for all the three temperatures in which indicated that the RWP-modified bitumen is higher strain susceptibility but become less steep when the addition of the RWP decreased irrespective of RWP types. Besides that, the result showed from the fatigue lines is parallel with the absolute value of parameter 'B' in which RWP-modified binders have higher 'B' value and this yield greater slope of fatigue line. This happen because of the RWP-modified bitumen tend to produce brittle failure when subjected to higher strain level. Saboo and Kumar (2016) added that the brittle failure occurred due to the higher stiffness at low temperature in which attributed to the crystalline nature of the plastomer itself. In 1999, study has been conducted by Bahia et al. (1999) found that

modified binders were vary in strain sensitivity and some binders are more strain dependent due to its microstructure.

Moreover, the neat bitumen exhibited remarkable behaviour at all temperatures. It was found that at low strain level, the neat bitumen had lowest fatigue lives but as the level of strain increased, the rate of decrease in fatigue lives was lower than the modified binders. This means that the neat bitumen was less susceptible to strain amplitude at all strain level.

Moreover, the prediction of fatigue lives could be done at the two strain level namely 2.5% and 5% in which the binder strain for strong pavements with thicker asphalt layer (>4 inches) is represented by the strain level of 2.5% meanwhile the strain level of 5% represent the strain of the binder of weak pavement with thinner asphalt layer (Jafari & Babazadeh 2016). As shown in Table 5.4 at both strain levels, the RWP modification give better fatigue lives particularly at high modification level.

As illustrated in Figures 5.21 to 5.23, adding RWP as modifier could improve the fatigue life of the base binder. The improvement in fatigue resistance is more pronounced at modification level of 7% especially for RWP of LDPE and HDPE. Thus, further investigation on comparison of higher level modification binders with binder that have similar penetration grading as well as a SBS-modified bitumen is investigated and discussed in the next chapter.

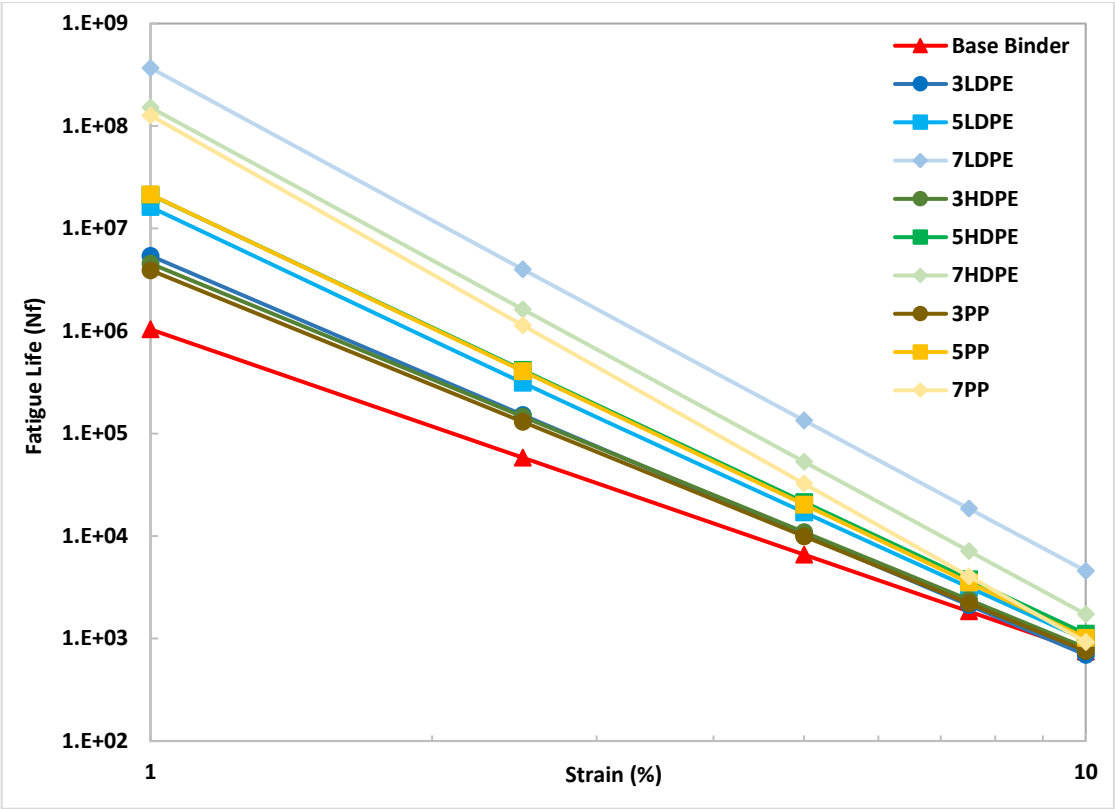


Figure 5.21: Fatigue life against strain levels at temperature of 15°C

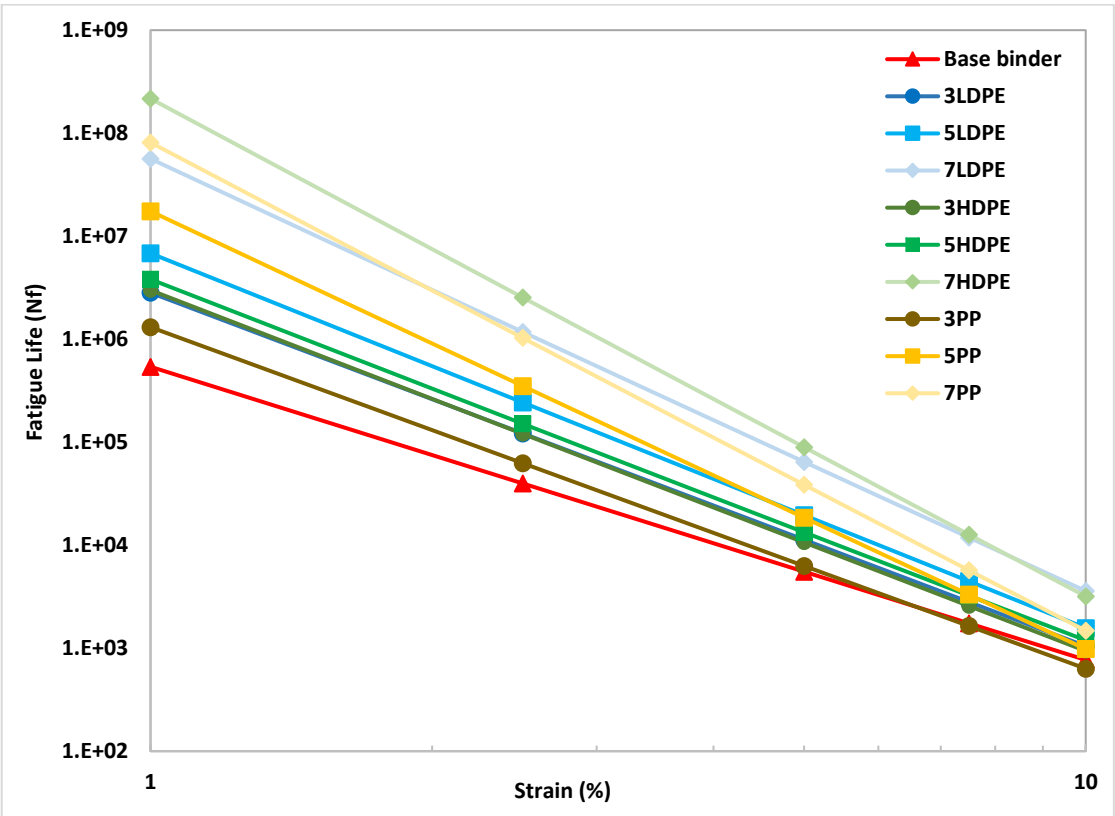


Figure 5.22: Fatigue life against strain levels at temperature of 20°C

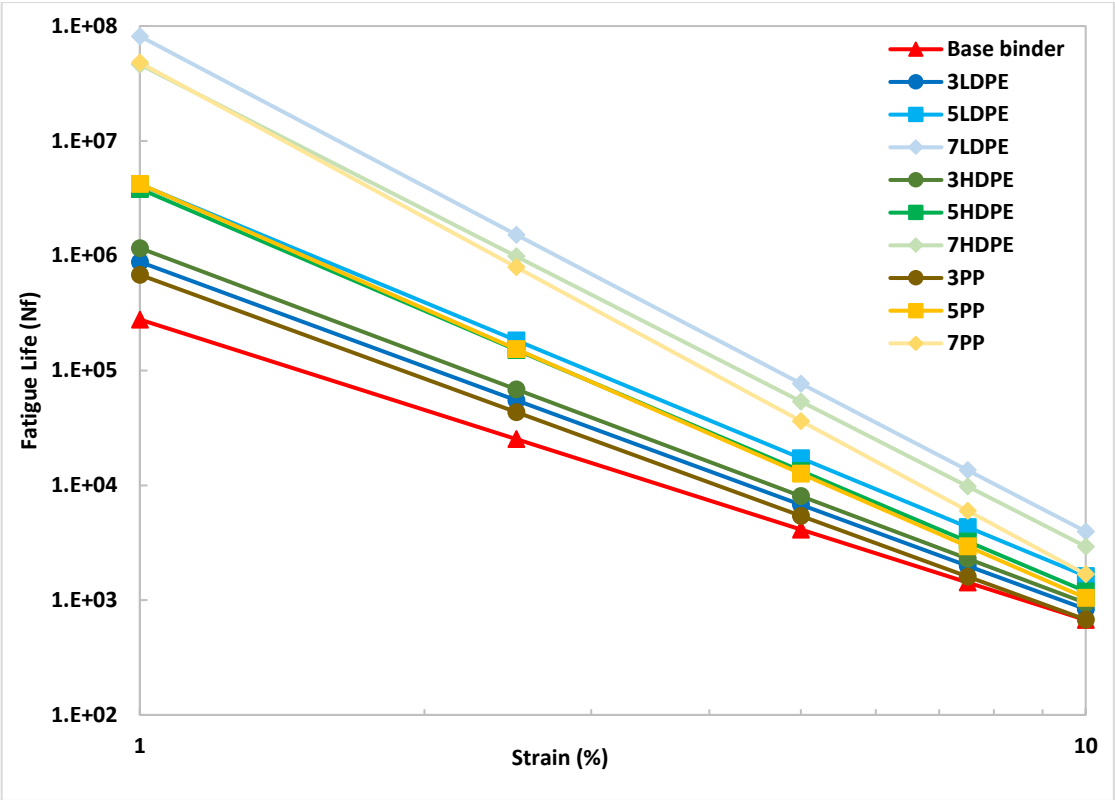


Figure 5.23: Fatigue life against strain levels at temperature of 25°C

Table 5.4: The Fatigue life of all binders at strain level of 2.5% and 5% for all tested temperatures

Binder	Fatigue life, N_f					
	2.5%			5%		
	15°C	20°C	25°C	15°C	20°C	25°C
Base binder	58,207	39,765	25,251	6,583	5,531	4,117
3LDPE	151,900	121,691	55,005	10,228	11,284	6,766
3HDPE	146,458	120,482	68,514	10,904	10,634	8,040
3PP	130,326	62,522	43,387	9,937	6,285	5,413
5LDPE	312,996	243,153	182,997	17,014	19,538	17,220
5HDPE	416,513	151,084	151,084	21,422	13,341	13,341
5PP	406,749	351,109	153,587	20,310	18,479	12,631
7LDPE	4,008,279	1,175,415	1,523,298	134,140	64,404	76,923
7HDPE	1,632,174	2,538,886	985,499	53,020	89,353	53,695
7PP	1,138,988	1,028,380	794,580	32,427	38,600	36,270

5.5.2 Double Edge Notch Tensile (DENT) Test

The double edge notched tension (DENT) test was used to evaluate the energy needed to fracture the binder in its ductile state by means of the essential work of fracture (EWF) method. The binders were aged with RTFOT and PAV before conducting the DENT test and the test was performed at testing temperature of 10^{o}C and 20^{o}C.

5.5.2.1 Key Assumptions Validation

A validation of the requirement or assumptions (as discussed in Chapter 3) need to be done for ensuring that the DENT test is valid and the EWF approach can be used for the analysis. For the first and second requirements, the plot of load versus displacement for different ligaments provides the indication. Figure 5.24 is the load-displacement curve of 3HDPE binder for different ligaments at a test temperature of 10^{o}C (other binders showed similar patterns and are provided in the appendix). It can be seen that the ligament is fully yielded as the load-displacement curve showed the flat shapes at maximum point of the load before tearing process takes place at the sudden drop of the curve. Besides that, Figure 5.24 depicted among different ligament lengths of binder tested, the similarity of these curves was good or in other words, the failure mechanism is similar for all ligament lengths. It means that all the binders experienced the same fracture processes which is stretching, yielding and tearing. In addition, the results also displayed that the test has good repeatability.

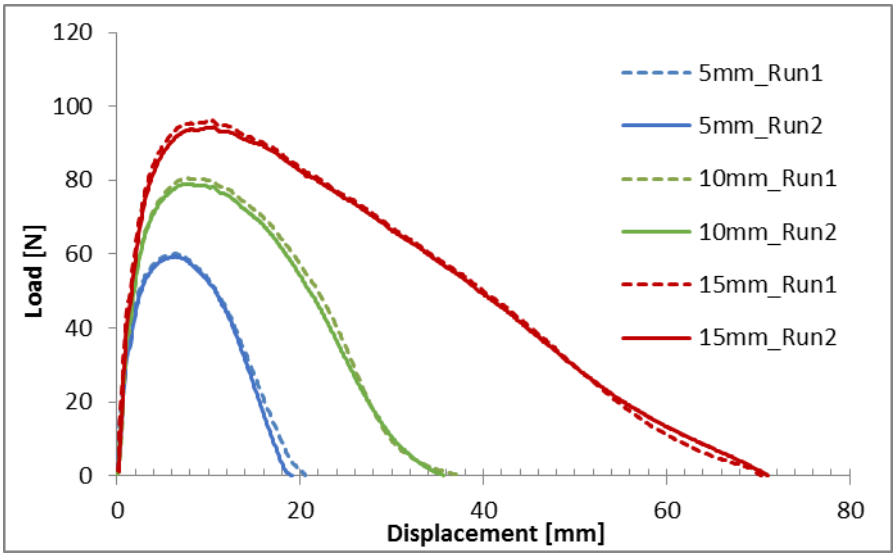


Figure 5.24: Load Displacement Curve for 3HDPE at temperature 10°C

The graph of the net section stress (σ_n) against the length of ligament (L) is used for validating whether the sample is in the plane stress condition or not. The plot of the net section stress as a function of the ligament length is displayed in Figure 5.25 for all binders at a test temperature of 20°C. Previous studies reported that the binder can be considered in the plane stress of failure regime if the net section stress of the binder increased as the ligament length is decreased (Andriescu et al. 2004; Subhy et al. 2017; Subhy 2017a). This condition is shown in Figure 5.25 for all binders used in the study at testing temperature of 20°C and a similar graph is displayed for tested temperature of 10°C. Thus, the third assumption is fulfilled at both testing temperatures.

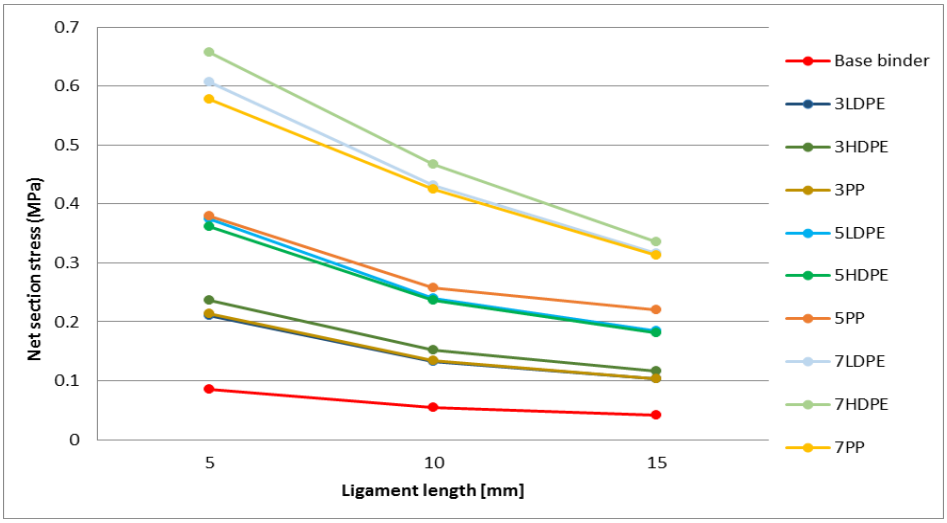


Figure 5.25: Graph of net section stress versus the ligament length at temperature of 20°C

5.5.2.2 The Curve of Load Versus Displacement

The force displacement curve acquired from DENT test at testing temperature of 10 and 20°C for base binder and binder blended with 3%, 5%, and 7% of LDPE is shown in Figures 5.26 and Figure 5.27, respectively (HDPE and PP displayed similar curves). This curve could give an idea about total energy stored in a binder (Singh & Girimath 2016). As illustrated, the addition of RWP into base binder increased the maximum force and at the same time decreased the displacement at failure when the ligament length increased. This phenomenon became more significant with an increase in the amount of RWP added, indicating the RWP-modified bitumen is stronger but less flexible and tend to fracture early, thus has poorer fracture properties. It happens due to rigidity of the plastomer nature of the RWP. Singh and Girimath (2016) and Subhy et al. (2017) noted that the stiffer binder could reduce the ductile nature of the bitumen.

The force displacement graph for all binders showed a clear one yielding point (one peak). However, the previous study done by Singh and Girimath (2016) found two peaks for SBS (3.5%) modified bitumen due to the strain hardening of the SBS polymer

(Subhy et al. 2017). Zoorob et al. (2012) stated that the presence of strain hardening was depending on the arrangement and orientation of the structural unit of the polymer.

Moreover, when the testing temperature is increased, there is a reduction in the maximum force point but the failure displacement is increases. This shows that higher temperature will soften the binder and make the modified binder more able to stretch. It can be concluded that higher maximum load peak with early failure of a sample is exhibited by the RWP-modified bitumen due to the higher stiffness of these binders.

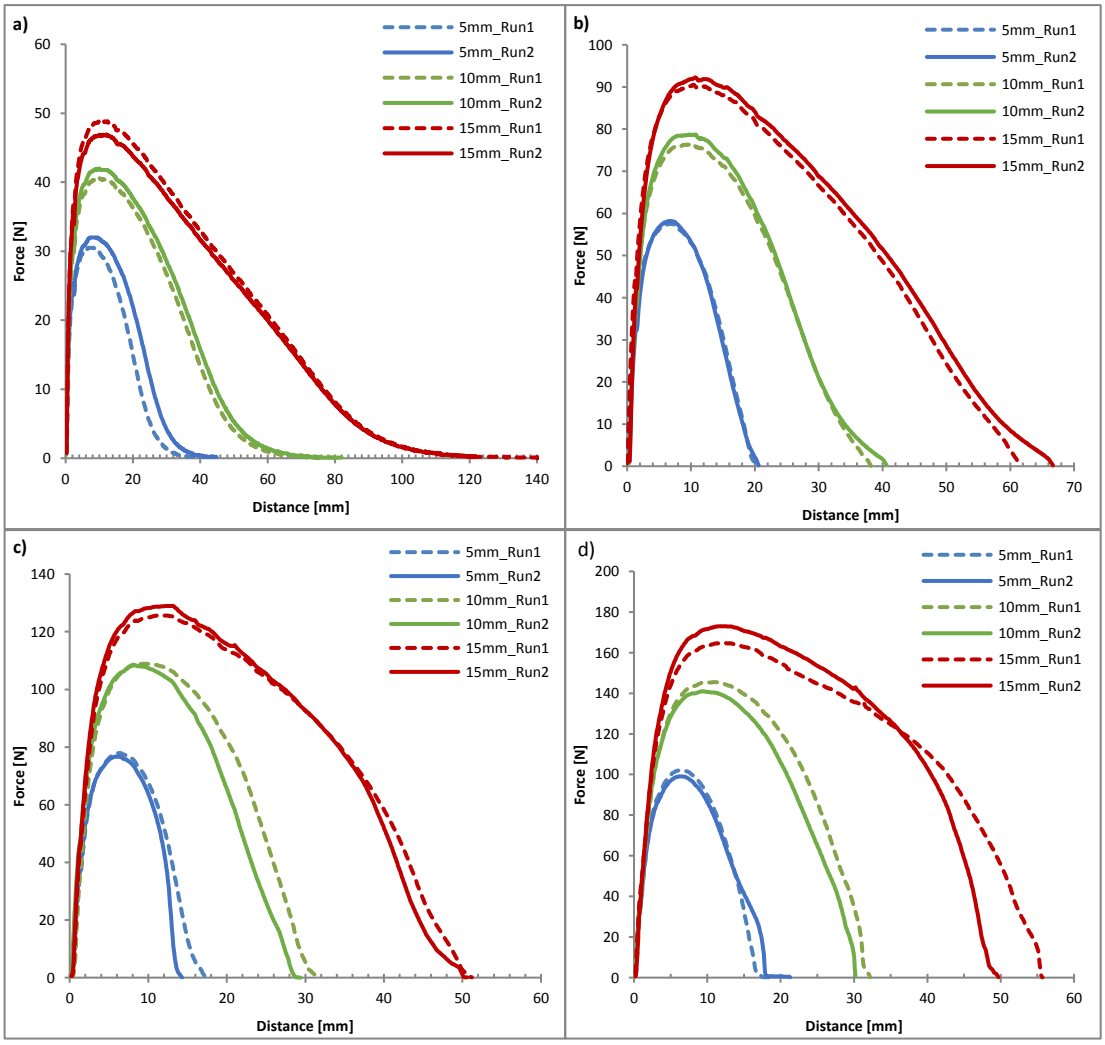


Figure 5.26: Force displacement curve at temperature of 10°C for (a) Base binder, (b) 3LDPE, (c) 5LDPE, (d) 7LDPE.

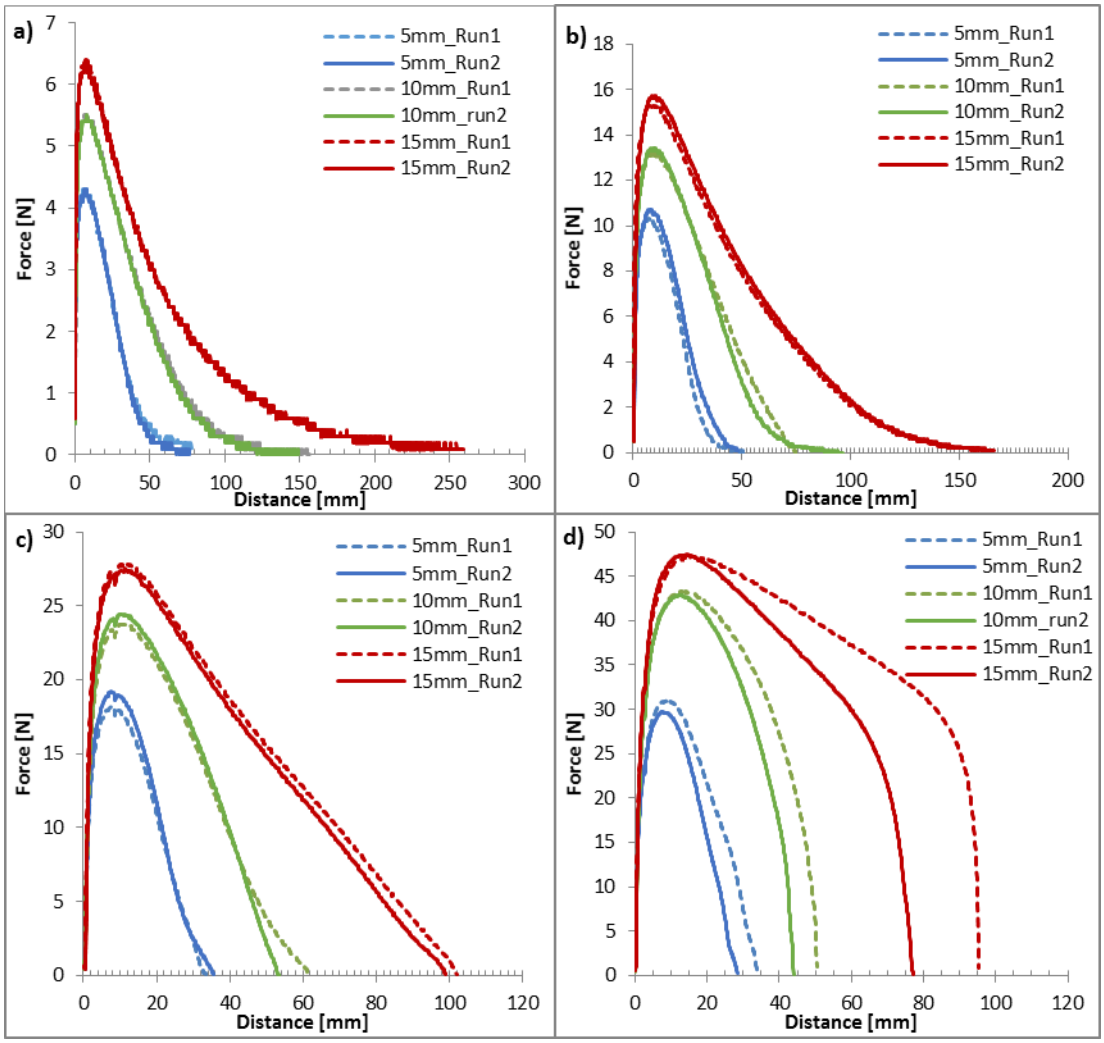


Figure 5.27: Force displacement curve at temperature of 20°C for (a) Base binder, (b) 3LDPE, (c) 5LDPE, (d) 7LDPE

5.5.2.3 Effects of RWP on Force Capacity and Displacement at Failure

Figures 5.28 and 5.29 show the comparison of the maximum load carrying capacity and displacement at failure for base binder and all RWP-modified binders at temperatures of 10 and 20°C, respectively. Schapery and Park (1999) stated that binders with high viscosity tend to have more load carrying capacity and failed at a shorter length. Similar behaviour occurred in this study for all RWP-modified binders that are having higher viscosity values compared to the base binder. According to both figures, in term of maximum load carrying capacity, the RWP-modified binders at both

testing temperatures displayed a significant increment with RWP-modified binders compared to the base binder for both testing temperatures. This indicates that the RWP-modified binders could withstand more loading during crack initiation process. All three types of RWP-modified binders presented almost similar values of maximum load capacity for each amount of modification at both testing temperatures.

Moreover, the decrease in displacement when failure occurred is more pronounced with addition of RWP and no specific trend can be observed due to the types of RWP used for each modification. At testing temperature of 10^{o}C, the displacement decreased significantly when the addition of RWP regardless of RWP types for 3 to 5% and as the amount of RWP increase to 7%, no further reduction in the displacement is perceived. Conversely at testing temperature of 20^{o}C in which the content of RWP added increased makes the displacement at failure decreased. Thus, these two parameters were not dependent on the RWP types but strongly dependent on the testing temperature as the temperature increased and the amount of the RWP added into the base binder.

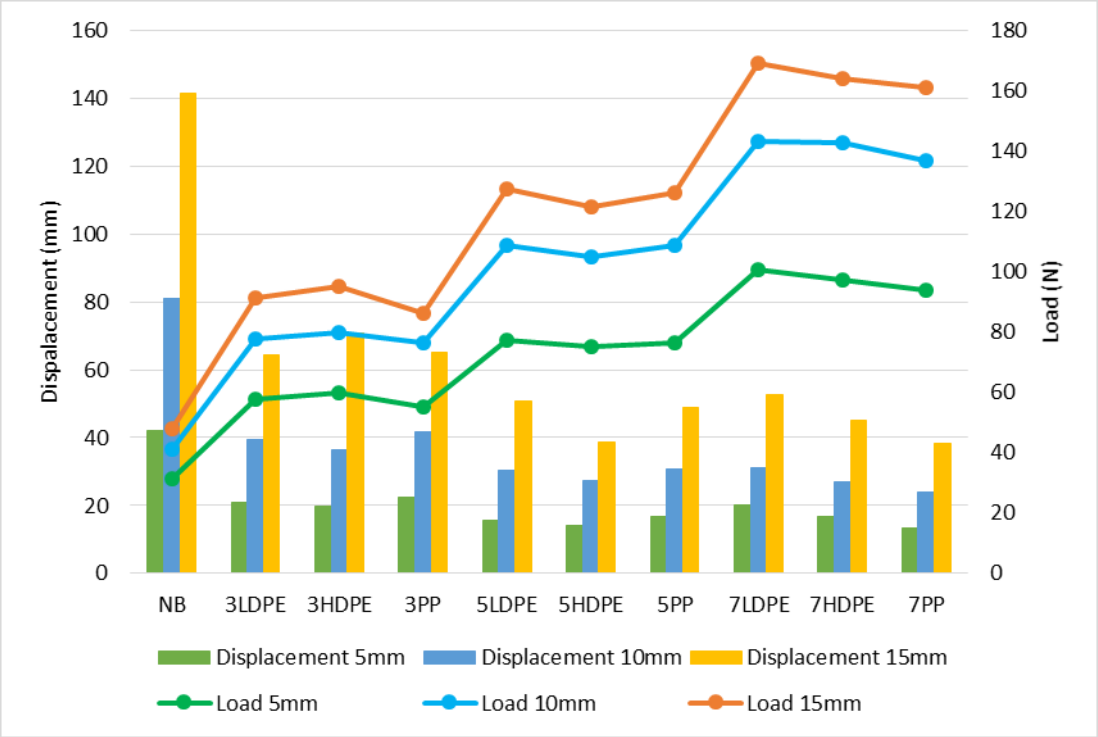


Figure 5.28: Force capacity and displacement at failure of RWP-modified bitumen at testing temperature of 10°C

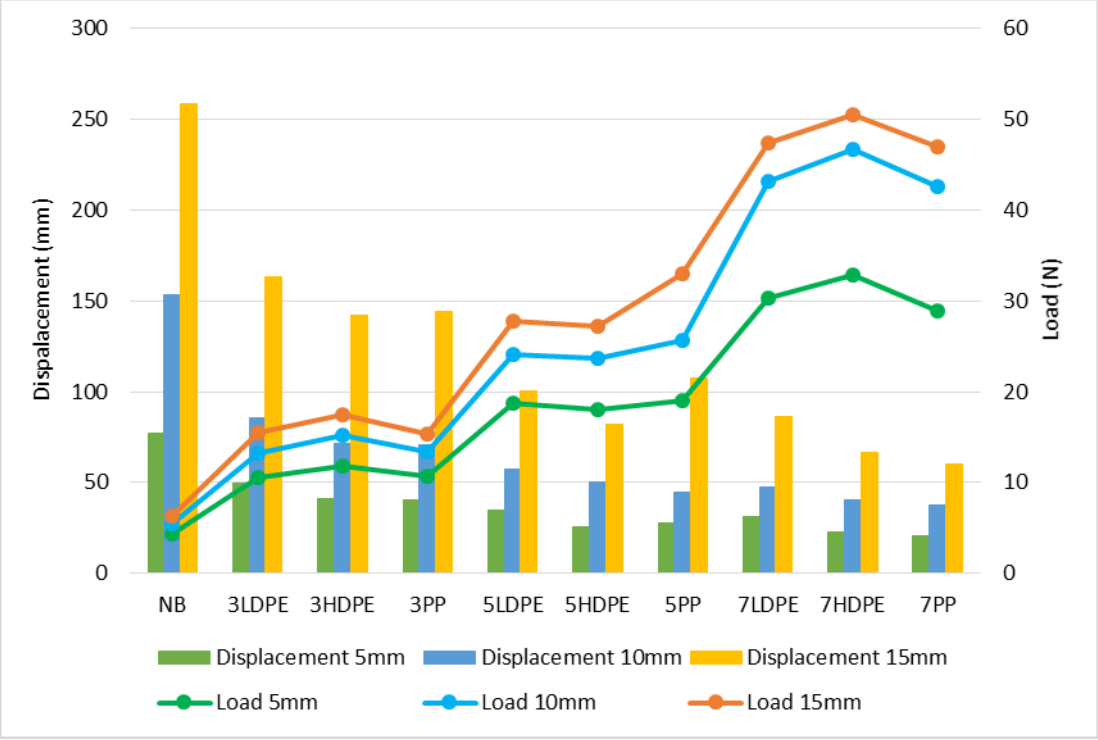


Figure 5.29: Force capacity and displacement at failure of RWP-modified bitumen at testing temperature of 20°C

5.5.2.4 Effects of RWP on Specific Total Work of Failure

By using the area under the load-displacement curve, the specific total work of fracture (W_t) is obtained for each ligament length as shown in Figure 5.30 for all RWP-modified binders. It can be seen that there is a significant increase in specific work of fracture for the binders with the addition of RWP. The linear regression analysis in Figure 5.32 shows higher R^2 values (the fitting procedure is acceptable) which indicates that the assumptions of the essential work of fracture method is satisfied (Bahia et al. 2001; Jafari & Babazadeh 2016).

According to the plot of the specific total work of fracture, the linear fitting procedure of the straight line yields the specific essential work (w_e) and the plastic work of fracture (βw_p) that can be determined from the intercept and slope of the line, respectively. Figure 5.31 presents the value of the intercept denoted as w_e and the slope of the line denoted as βw_p for base binder and RWP-modified binders at testing temperatures of 10 and 20°C. In order to have better fatigue cracking resistance, the binder should have higher values of w_e and βw_p (Schapery & Park 1999). The value of w_e is considered as a material property and a vital parameter for fatigue cracking in which this parameter is related to the damage process (Bahia et al. 1999; Schapery & Park 1999; Zhou et al. 2012; Jafari & Babazadeh 2016) while the βw_p is involved in the deformation outside the fracture zone of the material and thus, less important in the fatigue cracking (Schapery & Park 1999). However, Andriescu et al. (2006) stated that the βw_p value could be used for clarifying the discrepancies in the binder performance that are having equal w_e values.

It can be seen that the w_e and βw_p values increased when blending the base binder with RWP. This indicates that adding RWP into the base binder increases the

energy needed to fracture the samples due to higher stiffness compared to base binder and thus, good fracture resistance should be expected for RWP-modified bitumen. The higher values of w_e and βw_p were reported for 7% RWP-modified binders regardless of RWP types for both testing temperatures. Moreover, when the temperature of testing increases, a reduction in the values of w_e and βw_p is observed. However, the parameter of w_e and βw_p were unable to evaluate fatigue cracking behaviour of the binder compared to the critical tip opening displacement (CTOD) parameter. Therefore, the evaluation of CTOD parameter on RWP-modified bitumen will be described in the next section.

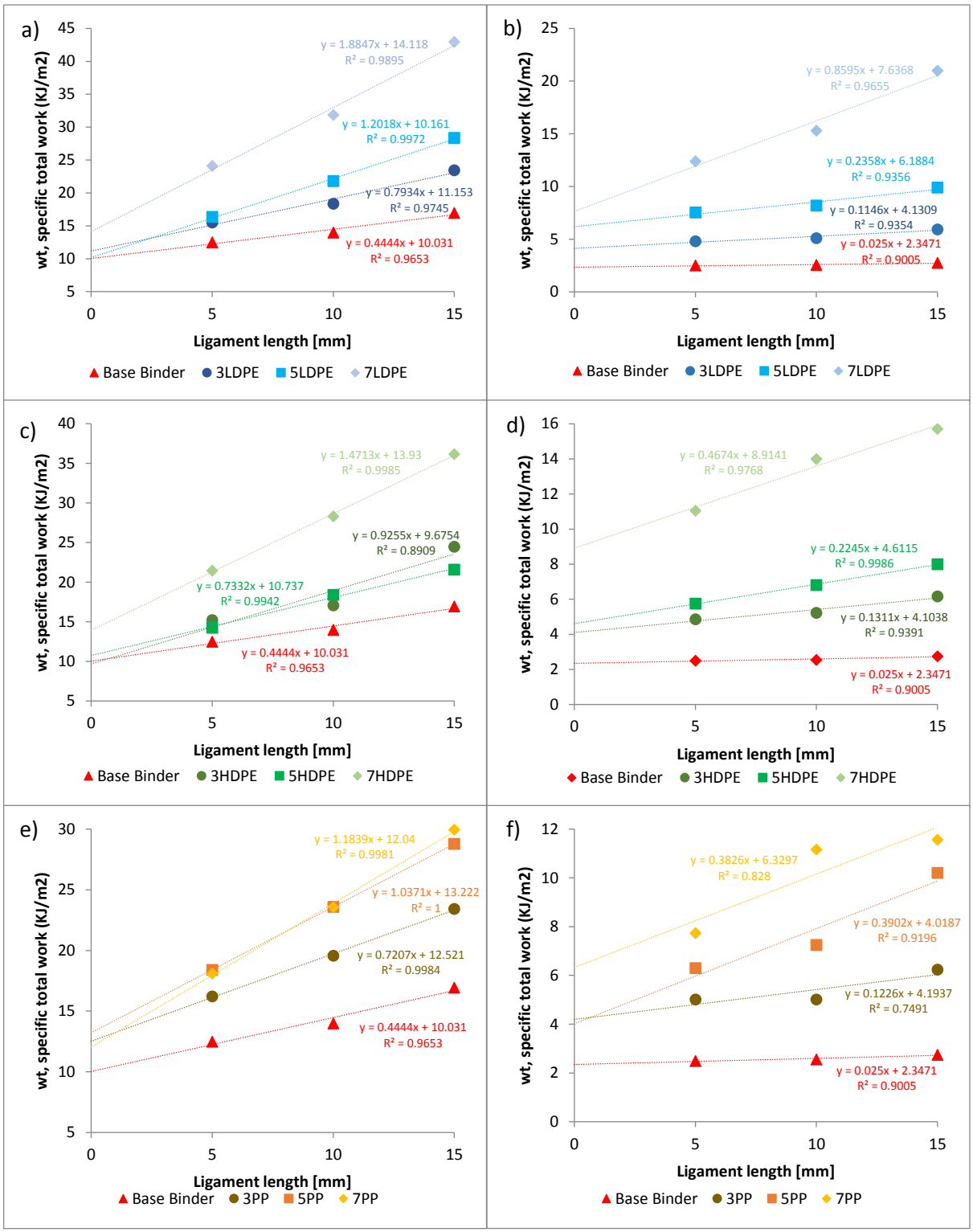
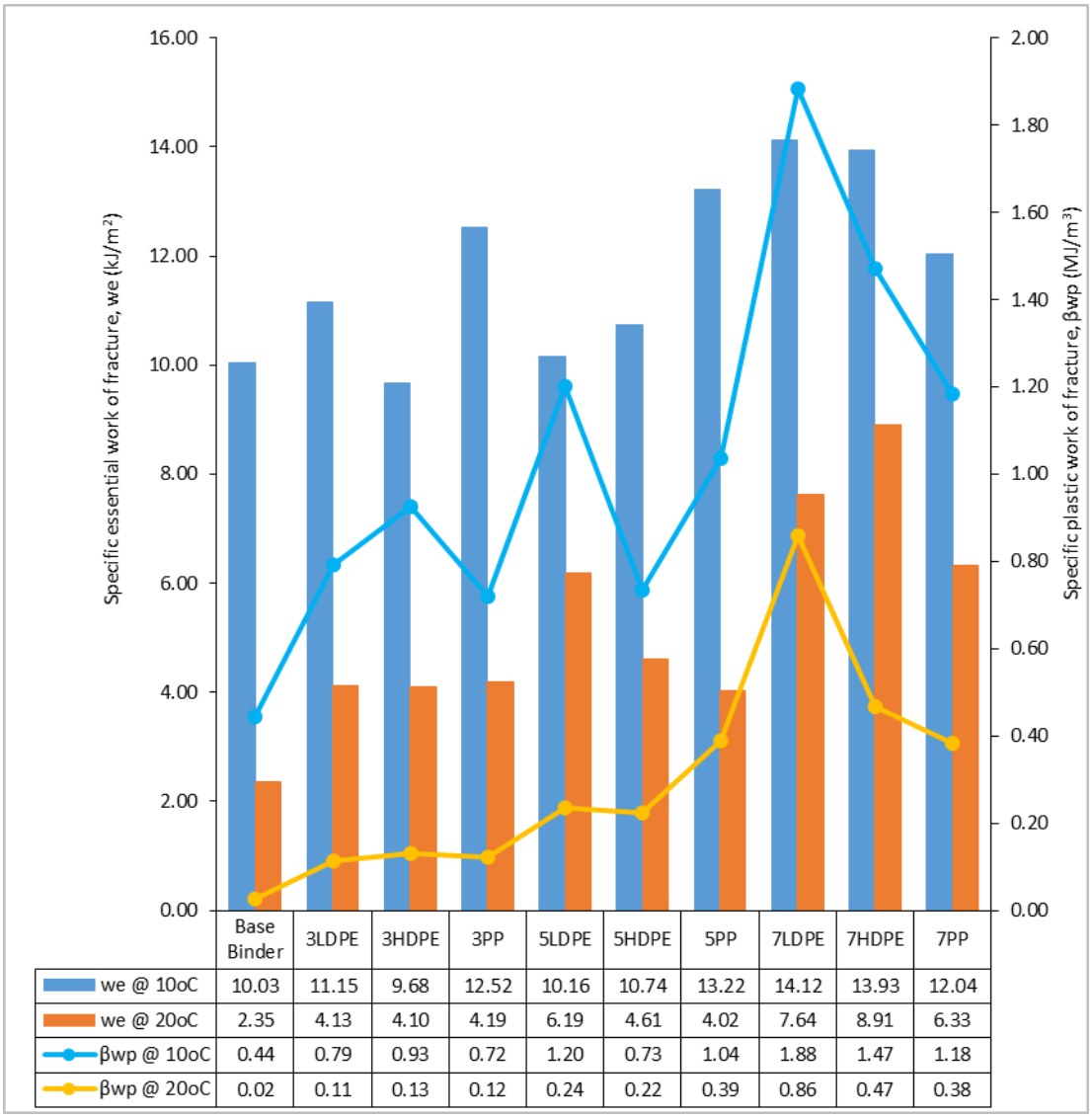


Figure 5.30: Specific total work of fracture for base binder with additional of a)LDPE at testing temperature 10°C, b) LDPE at testing temperature 20°C, c) HDPE at testing temperature 10°C, d) HDPE at testing temperature of 20°C, e)PP at testing temperature 10°C, and f) PP at testing temperature 20°C



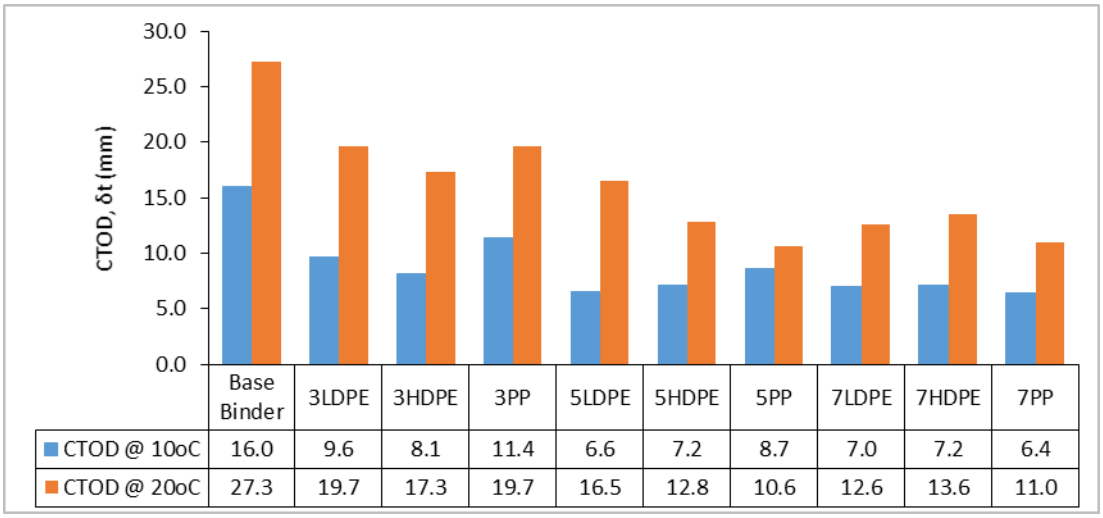
	Base Binder	3LDPE	3HDPE	3PP	5LDPE	5HDPE	5PP	7LDPE	7HDPE	7PP
we @ 10oC	10.03	11.15	9.68	12.52	10.16	10.74	13.22	14.12	13.93	12.04
we @ 20oC	2.35	4.13	4.10	4.19	6.19	4.61	4.02	7.64	8.91	6.33
βwp @ 10oC	0.44	0.79	0.93	0.72	1.20	0.73	1.04	1.88	1.47	1.18
βwp @ 20oC	0.02	0.11	0.13	0.12	0.24	0.22	0.39	0.86	0.47	0.38

Figure 5.31: The specific essential work of fracture and the plastic work of fracture for all RWP-modified binders at testing temperature of 10 °C and 20°C

5.5.2.5 Effects of RWP on Critical Tip Opening Displacement (CTOD) Value

The comparison of CTOD values for base binder and RWP-modified binders at testing temperatures of 10 and 20^{o}C is displayed in Figure 5.32. Higher CTOD value is desired for a binder to have a better fatigue resistance which indicates that the strain tolerance of the binder in the presence of a crack in ductile failure is higher, thus more resist to fracture (Johnson 2010; Jafari & Babazadeh 2016). Decrease in the CTOD value is significantly shown by the RWP modification of base binder at both testing temperatures and as the amount of RWP added is increased, the CTOD values become lower as compared to base binder. Besides that, no specific trend could be observed among the types of RWP used for the reduction for each amount of addition. This indicated that the fracture resistance of the RWP-modified binders is poor. The reduction of CTOD value for RWP-modified binders may be due to the higher stiffness of the modified binders. In fact, the base binder used in this study is a soft binder and less viscous at low temperature, hence making it unable to withstand higher load as this binder has low stiffness value compared to RWP-modified binders. Nevertheless, higher stiffness of RWP-modified binders makes them stronger but less able to stretch due to the rigid characteristics of the RWP itself. Moreover, the strain tolerance of the RWP-modified binders at different temperatures could be described from the result of CTOD value at two testing temperatures. It can be inferred that by considering its strain tolerance at temperatures of 10 to 20^{o}C, the RWP-modified binders exhibited increased strain tolerance but still having worst fracture characteristics compared to base binder as evident of higher CTOD value of the base binder. Generally, Jafari and Babazadeh (2016) stated that the binder tends to have lower stiffness and able to stretch more if the temperature increases.

In can be concluded that the RWP-modified binders have small strain tolerance and thus, are more susceptible to fracture but it could withstand higher strain levels due to the higher viscosity value that make it stiffer compared to base binder as evidence of higher load carrying capacity. Due to the higher stiffness value, the RWP-modified binders need more energy to create two new fracture surfaces and thus, makes the value of parameter w_e higher than the base binder.



	Base Binder	3LDPE	3HDPE	3PP	5LDPE	5HDPE	5PP	7LDPE	7HDPE	7PP
CTOD @ 10oC	16.0	9.6	8.1	11.4	6.6	7.2	8.7	7.0	7.2	6.4
CTOD @ 20oC	27.3	19.7	17.3	19.7	16.5	12.8	10.6	12.6	13.6	11.0

Figure 5.32: The CTOD values for base binder and RWP-modified binders at testing temperature of 10 °C and 20°C

5.6 Rutting

At high pavement temperature conditions which are more than 40°C whereby the binder tends to become softer and more viscous, the major failure mode exhibited by the flexible pavement is rutting or permanent deformation. Rutting appears as a result of the increasing number of traffic load repetitions which cause the plastic deformation of the materials of each layer in the pavement structure. So, the material properties that is related to the performance test at higher temperature were evaluated by means of the Multiple Stress and Creep Recovery (MSCR) test.

5.6.1 Multiple Stress and Creep Recovery (MSCR)

In order to evaluate the resistance of binder to rutting, the advanced test which is the MSCR test was used at test temperatures of 40, 60 and 70°C using the DSR. All the binders have been aged for short time by means of RTFOT as the earliest rutting may occurred after the manufacturing process. According to BS EN 16659:2015, the MSCR test covers the determination of non-recoverable creep compliance and percent recovery of bitumen.

5.6.1.1 Non-recoverable Compliance (J_{nr})

Tables 5.5 presents the non-recoverable creep compliance (J_{nr}) values of the base binder and RWP-modified binders at different temperatures and stress level that is obtained from MSCR test. The higher J_{nr} values indicates that binder is susceptible to rutting or in other word, the tendency of the binders to return to its original shape is less once the load is removed (Hafeez et al. 2014). According to the latest AASHTO M 332-14 specification, the J_{nr} values at 3.2 kPa stress level according to different traffic loads as shown in Table 5.6.

It can be seen that the J_{nr} values for base binder were less than 4.5 kPa^{-1} at temperature of 40°C for all stress levels. However, as the temperature increased to 60 and 70°C, an increase in the J_{nr} values exceeding the limit is observed for all stress level, meaning that the base binder is prone to rutting at these two temperatures. From the table, it showed a reduction in J_{nr} values with the addition of the RWP into base binder and become more significant as the RWP content is increased for all types of RWP. Thus, it can be noted that the utilisation of RWP for modifying base binder would give a beneficial effect on the rutting resistance.

At temperatures of 40 and 60°C, the value of J_{nr} for all RWP-modified binders remain within the limits of AASHTO even at higher stress level (12.8 kPa). When the testing temperature increase up to 70°C at all stress levels, all RWP-modified binder with 3% modification level showed J_{nr} values higher than the limits (more than 4.5 kPa^{-1}). As the amount of RWP increased particularly at stress level of 0.1 and 3.2 kPa (except for 5LDPE and 5HDPE-modified binder), the J_{nr} value confined to the limits at standard heavy traffic and this value become worst as the stress level increase to 12.8 kPa. This means that when the pavement design temperatures are higher than 70°C and below stress level of 12.8 kPa, the modification of base binder with intermediate and high percentages (5 and 7%) of RWP does ensure acceptable rutting performance. Kataware and Singh (2017) stated that at high stress levels and temperatures below 60°C, the stiffness of the binder is enhanced by the polymer network and thus, exhibited lower J_{nr} values. They added that the significant increase in J_{nr} value at temperature higher than 60°C and higher stress level may due to the ability of the polymer network to hold the binder has weaken as the base binder become softer in which will result in a very high strain.

According to the specification, the base binder is unable to be used as a road pavement at high temperature climates due to higher J_{nr} value which indicates that this binder tends to have higher permanent deformation, lower modification level namely 3% could be used for standard traffic, 5% modification could be used for heavy traffic and higher modification level which is 7% could be used for very heavy traffic.

Overall, it can be concluded that the modification of base binder with RWP exhibited lower values of J_{nr} compared to base binder and thus, give a positive effect on the rutting resistance but this changed with temperature and stress levels. Besides

that, the rutting performance is dependent on the temperature and applied stress level as evidenced by an increment in the J_{nr} value with the increases of temperature and stress levels.

Table 5.5: J_{nr} of RWP-modified binders and base binder at all temperatures

Binder	Non-Recoverable Creep Compliance, Jnr (kPa^{-1})								
	40°C			60°C			70°C		
	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa
Base Binder	0.288	0.299	0.332	6.618	7.668	13.041	23.820	25.056	-
3LDPE	0.089	0.090	0.104	2.307	2.786	3.085	8.368	10.585	12.870
3HDPE	0.024	0.078	0.097	1.207	2.620	3.364	5.264	9.776	12.901
3PP	0.002	0.049	0.072	1.693	3.103	3.595	4.704	9.480	11.957
5LDPE	0.039	0.042	0.046	0.014	1.138	1.632	1.295	5.084	6.519
5HDPE	0.003	0.024	0.039	0.005	0.980	1.675	0.258	4.502	7.275
5PP	0.001	0.018	0.044	0.001	0.868	2.319	0.002	3.139	6.512
7LDPE	0.002	0.013	0.018	0.007	0.336	0.728	0.202	1.341	4.606
7HDPE	0.0003	0.0003	0.003	0.006	0.404	0.906	0.075	1.118	10.073
7PP	0.0002	0.0003	0.010	0.001	0.408	1.028	0.0004	0.845	6.948

Table 5.6: Traffic level according to J_{nr} (AASHTO M 332, 2014)

Traffic Level	Number of ESALs	Maximum Value of J_{nr} at 3.2 kPa (kPa^{-1}
Standard (S)	< 10 x 10^6 and 70 km/h	4.5
Heavy (H)	10 x 10^6 - 30 x 10^6 or 20 – 70 km/h	2.0
Very Heavy (V)	> 30 x 10^6 or < 20 km/h	1.0
Extreme (E)	>30 x 10^6 and <20 km/h	0.5

5.6.1.2 Stress Sensitivities of Binders

Apart from being an indicator for rutting resistance, the non-recoverable creep compliance could be used as an indicator for the sensitivity of bituminous binder to permanent deformation and its stress dependence for modified bitumen (D'Angelo 2009a). Figures 5.33, 5.34 and 5.35 showed the graph of J_{nr} values at different applied stress levels for temperatures 40, 60^{o}C and 70^{o}C, respectively. Jafari and Babazadeh (2016) stated that the stress sensitivity was highly dependent on the temperature and stress level which as temperature or stress level increased, stress sensitivity of the binder increased as well.

In this study, it was found that the stress sensitivities of the binders are mainly dependant on the temperature and RWP content added into the bitumen. As depicted in all figures, the base binder and RWP-modified binders with low RWP content (3%) exhibited less sensitive to stress while bitumen modification with 5 and 7% of RWP showed higher stress susceptibility at all test temperatures. The base binder demonstrates the non-linear behaviour at stress levels higher than 12.8 kPa for test temperature of 40^{o}C and failure occurs when the applied stress is higher than 12.8 and 6.4 kPa at temperature of 60^{o}C and 70^{o}C, respectively because base binder is in the fluid form at these temperatures. Despite having inferior J_{nr} ranking, the base binder and low modification level showed significantly less stress sensitivity compared to other binders.

Delgadillo et al. (2006a) reported that some of the modified binders showed linear behaviour at the standard stress level up to 12.8 kPa. Increasing the stress sensitivity causes nonlinearity to increase and thus, more prone to rutting (Jafari et al. 2015). Nonlinearity behaviour was depicted by 3% modification level at stress level

higher than 12.8 kPa. Unfortunately, different linear behaviour was obtained for binders with 5 and 7% of RWP content in which these binders showed its nonlinearity as early as the stress level of 0.1 kPa at all temperatures (a repeat test is shown in Figure 5.34 for 5PP to indicate that such a behaviour is not caused from the experimental error). Hossain et al. (2015a); Hossain et al. (2015b) reported that usually, the strain response of polymer modified binder is nonlinear as the stress level increased because the polymer chains tends to rearrange significantly when the stress level is increased. Although the RWP-modified binders had higher stress sensitivity, the resistance to rutting of these binders is significantly better than the base binders due to the lower values of J_{nr}.

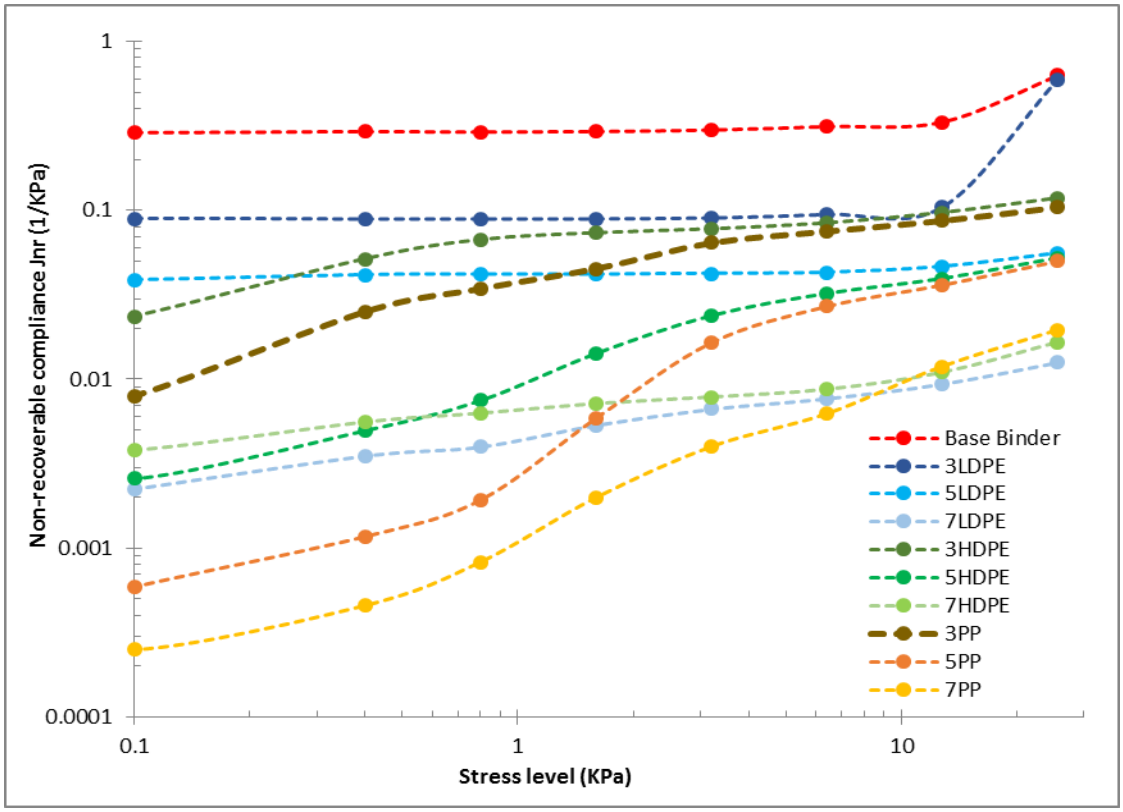


Figure 5.33: J_{nr} of RWP-modified binders and base binder at 40°C

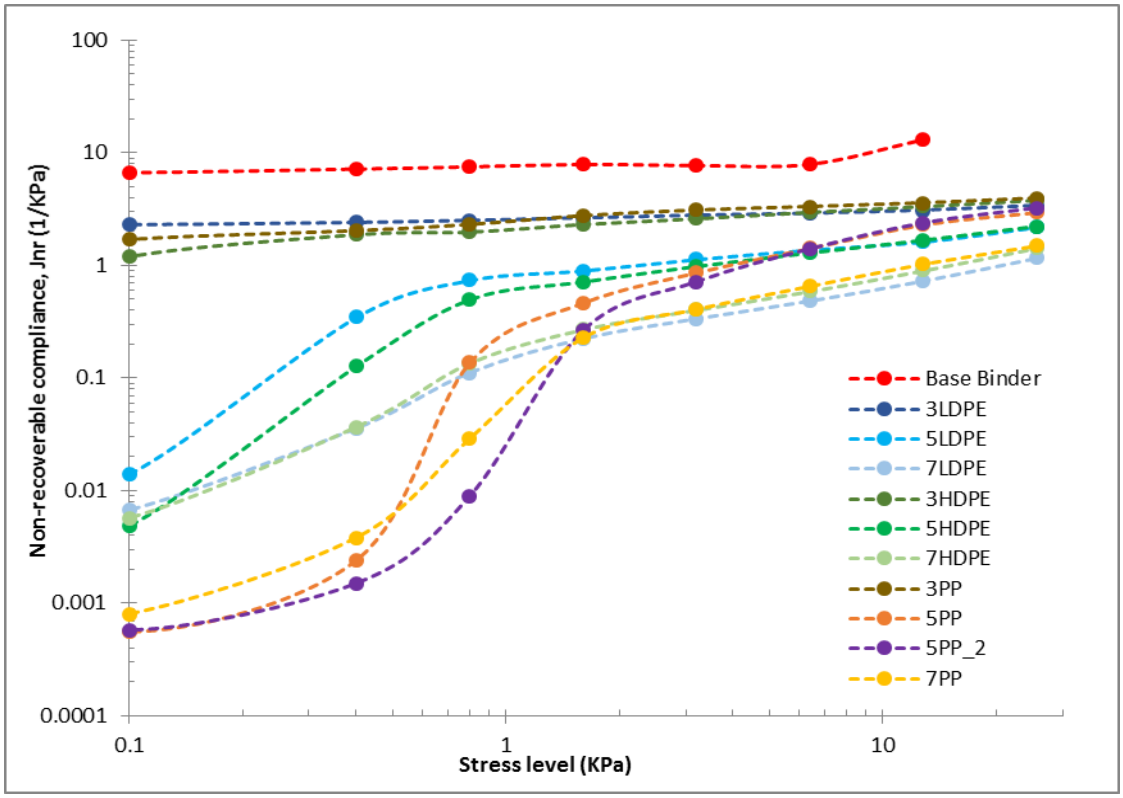


Figure 5.34: J_{nr} of RWP-modified binders and base binder at 60°C

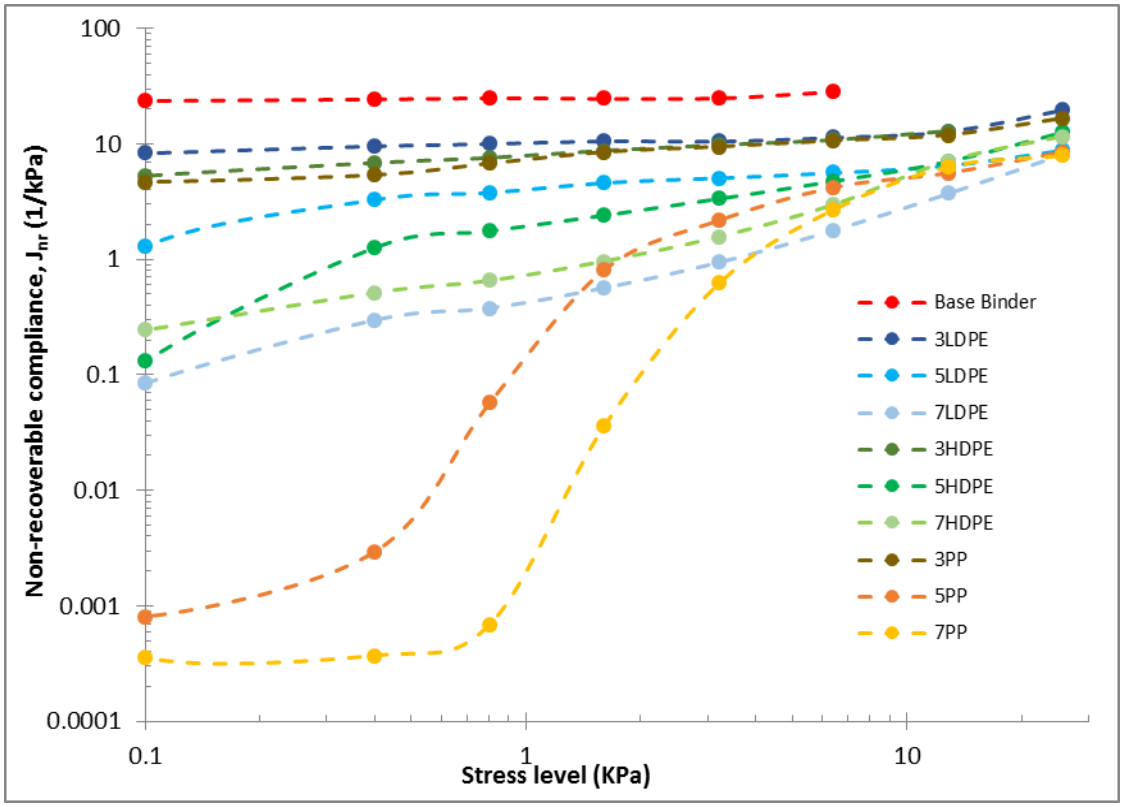


Figure 5.35: J_{nr} of RWP-modified binders and base binder at 70°C

5.6.1.3 Percentage of Recovery

At multi stress levels, the percent recovery (%R) is calculated to know the presence of elastic response and its stress dependence of bituminous binders. The percentage of recovery for all binders at different test temperatures and stress levels are shown in Table 5.6. The result showed that the addition of RWP into base binder causes an increase in %R value. Higher value of %R is desirable because it indicates that the binder can convalesce more amount of deformation and hence, resulting in low permanent deformations (Jafari & Babazadeh 2016). Based on the %R value, remarkable findings are obtained over a wide range of high pavement temperatures regarding the rutting performance of RWP-modified binders.

As can be seen in the Table 5.7, the addition of RWP showed better results in %R value at all test temperatures. As expected, the amount of RWP added affects the %R as the percentage of RWP increased, the significant increase in %R value is recorded but these values demonstrated a reduction when the temperature and stress levels increased. However, among all the RWP-modified binders, no specific trend could be observed, meaning that the percentage of recovery is strongly dependent on the amount of the RWP used to modify the base binder rather than the type of RWP used.

However, some of the %R displayed negative values at some temperatures and stress levels where this value is obtained if the strain response is increasing in the unloading phase (Soenen et al. 2013). Jafari and Babazadeh (2016) stated that these negative elastic recovery values can be related to the large strains due to a combination of high stress, high temperature and low modification level. In certain conditions of this test, some rheometers are unable to unload stress applied as fast as is requested. The strain response therefore increases in the unloading portion which

in the absence of load and for the high penetration grade bitumen having almost no recovery, results in a very small increase in the strain curve which is adequate to make the total recovery a negative value (Soenen et al. 2013; Jafari & Babazadeh 2016).

Table 5.7: Percent of recovery value for all binders

Binder	Recovery, R (%)								
	40°C			60°C			70°C		
	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa
Base Binder	15.5	12.3	5.0	8.5	0.0	-1.1	0.9	-1.3	-
3LDPE	29.0	25.2	15.9	12.6	1.6	0.3	12.1	-0.2	-0.8
3HDPE	75.8	29.4	17.7	40.2	2.2	0.3	8.8	0.0	-0.8
3PP	96.8	39.8	18.4	26.5	1.5	0.2	17.3	0.2	-0.6
5LDPE	43.0	37.3	29.0	92.8	10.1	2.7	26.3	-0.2	0.0
5HDPE	92.3	50.4	30.8	96.5	7.6	1.6	58.0	1.3	-0.2
5PP	98.1	63.0	30.3	99.0	6.8	1.1	96.1	2.2	0.1
7LDPE	92.1	60.3	47.8	95.1	28.5	9.2	61.0	14.8	1.4
7HDPE	94.0	94.6	71.9	93.9	13.3	3.1	57.6	8.6	-0.1
7PP	98.0	96.8	51.1	98.6	10.7	2.5	97.3	4.3	0.2

5.7 Summary

1. The result of conventional tests that encompassed penetration, softening point and viscosity tests have shown that the addition of RWP into base binder resulted in increased hardening and viscosity of the binder regardless of the types of thermoplastics used and became more significant as the amount of the RWP added is increased. Moreover, the modified binders may be able to be used on the pavement that has a wider temperature range as a result of the higher PI value particularly for 7% of RWP added compared to the base binder. Thus, the basic

properties of the binder are affected by the modification of base binder with RWP and highly dependent on the concentration of the RWP added rather than the RWP types used.

2. The effect of adding RWP into base binder is almost the same with the base binder at higher frequencies levels or low temperatures. The master curves and Black diagrams of unaged binders showed that the RWP-modified binders may help rutting resistance and are more pronounced as the content of RWP increased.
3. After going through short-term and long-term ageing, the complex modulus and phase angle master curves for all RWP-modified binders depicted almost the same increase and decrease with ageing respectively which is equivalent to an increase in the stiffness and elastic response after ageing, thus makes the binders behave more solid-like.
4. For all RWP-modified binders, the increment in ageing index value of complex modulus is lower at a temperature of 25^{o}C and higher at 65^{o}C. While for the phase angle, the reduction of the phase angle is consistence at 25^{o}C but in contrast for a temperature of 65^{o}C. However, the phase angle values become lower than the unaged binders as the RWP-modified binders went through PAV ageing. It can be concluded that the RWP-modified binders may have solid-like properties as evidenced by the increase in the stiffness and elastic response with ageing.
5. Based on result of the LAS test at temperatures of 15, 20 and 25^{o}C, the modification of base binder with RWP could offer positive improvement for the fatigue lives in which the higher modification level of base binder with RWP especially for LDPE and HDPE had better fatigue resistance than other modification levels. Nevertheless, the base binder tends to have similar fatigue

resistance with the RWP-modified binder at tested temperature of 15^{o}C and had lower strain susceptibility compared to RWP-modified binders at all tested temperatures.

6. From the DENT test:
    - The addition of RWP changes the trend of the load displacement curves as the amount of RWP added is increase. Adding more RWP into the base binder (more than 7%) will probably make the appearance of a second peak load more significant.
    - The RWP-modified binders had small CTOD value and thus are more susceptible to fracture but it could withstand higher strain levels due to higher viscosity values that make it stiffer compared to base binder as evidenced by higher load carrying capacity but lower displacement at failure. Due to the higher stiffness value, the RWP-modified binders need more energy to create two new fracture surfaces which makes the value of both the specific essential work of fracture and the plastic work higher than the base binder.
7. The result of J_{nr} and R values of RWP-modified binders were found to be lower and higher respectively compared to the base binder indicating less susceptibility to rutting. As the amount of RWP increased, the modified binders exhibited better rutting performance. These parameters were highly affected by the increase in temperature and stress levels of the test.

The positive effect of RWP additive on improving the mechanical properties of base binder is highly dependent on the amount of additive added and more pronounced for the high modification level which is 7% rather than the types of RWP additive used.

Thus, 7% of RWP-modified binders for all types of RWP additives will be used for further investigation.

CHAPTER 6: HIGHER LEVEL MODIFICATION OF RWP-MODIFIED BITUMEN

6.1 Introduction

Based on the experimental works done in Chapter 5, it has been shown that a 7% modification of base binder with RWP provided an improvement with respect to the mechanical properties and performance of the binders. Thus, only 7% RWP-modified binders were used in this chapter for further comparison.

Another two conventional binders were tested and compared with the 7% RWP-modified binder consisting of a 40/60 penetration grade bitumen (written as control binder, CB) which represents the similar penetration grade of high modification (7% RWP-modified binders) of base binder and a SBS-modified bitumen representing a ready-made polymer modified bitumen (PMB).

This chapter deals with the comparison between the higher modification level, base binder, control binder and the ready-made PMB which is the SBS-modified bitumen in term of mechanical properties, rheological properties, fatigue cracking, fracture resistance and rutting.

6.2 Mechanical Properties

6.2.1 Penetration and Softening Point test

Table 6.1 shows the result of penetration and softening point for higher level modification of RWP-modified binders, base binder, control binder and SBS-modified bitumen. In comparison between SBS-PMB and 7% RWP-modified binders, the SBS-PMB has higher penetration and lower softening point values with 87 dmm and 84.2^{o}C, respectively. It indicates that the SBS-PMB is less stiff, so less temperature required for changing the phase from solid to liquid and thus lower softening point value.

For the similar penetration grade of bitumen, the conventional 40/60 penetration grade bitumen (noted as control bitumen) has 50% lower softening point value compared to the 7% RWP-PMBs. This means that the higher softening values of 7% RWP-PMB is caused by the modification and may be due to the stiffness of the RWP itself that will reduce the risk of permanent deformation in the field.

Table 6.1: Result of penetration and softening point test for 7% RWP-modified binders, base binder, control bitumen and SBS-modified bitumen

Binder	Penetration (dmm)	Softening Point (°C)
Base Binder (BB)	181	37.4
Control Binder (CB)	47	50.4
SBS PMB	87	84.2
7LDPE	42.2	104.5
7HDPE	42.4	119
7PP	44.5	120

6.2.2 Penetration Index (PI)

The temperature sensitivity of bitumen could be identified by using PI which is the susceptibility of the binder to the temperature is higher if the PI value is lower and more elastic behaviour (Nassar et al. 2012) and vice versa. Table 6.2 shows the PI value for all binders. It was found that the SBS-modified bitumen and all RWP-modified binders demonstrated higher PI values indicates that all modified bitumen are less temperature susceptibility. In comparison between base binder and all RWP-modified binders shows that the addition of RWP to modify base binder reduces the temperature sensitivity of base binder. Moreover, the PI index for the control binder is lower compared to the 7% RWP-modified binders with a value of -1.24 means that

RWP-modified binders had lower sensitivity against temperature and thus can be used at wide temperature range compared to control binder.

Besides that, the PI value can be used to evaluate the sensitivity of the binder against temperature by means of softening point-penetration chart. Figure 6.1 displays the chart of material property relating to the penetration and softening point for all binders. The material that is soft and temperature susceptible is normally located at the right and bottom of the plot while the harder materials and less temperature susceptible can be found at the left and top of the plot (Munera & Ossa 2014). It can be seen that all the polymer modified binders are hard and less sensitive against the temperature due to the polymer addition but in contrary for the base binder. Besides that, the control binder has similar behaviour regarding its temperature susceptibility with the base binder but as the penetration value is low, the binder is hard.

Table 6.2: Penetration index of 7% RWP-modified binders, base binder, control bitumen and SBS-modified bitumen

Binder	PI
Base Binder (BB)	-1.69
Control Binder (CB)	-1.24
SBS-MB	6.54
7LDPE	6.64
7HDPE	7.88
7PP	8.07

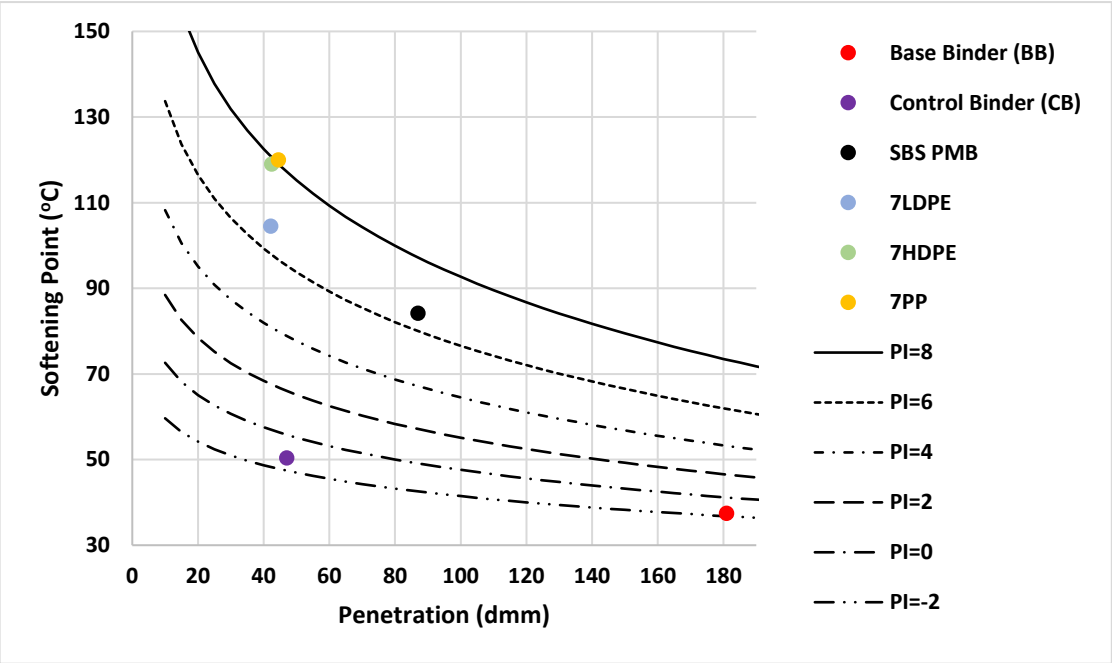


Figure 6.1: The material property chart for penetration and softening point

6.2.3 Viscosity test

The measurement of flow characteristics of binders can be obtained by means of viscosity test. For easily pumping and handling during the production of pavement, the binder must be adequately fluid at high temperatures (Santagata et al. 2012).

Although control binder and 7% RWP-MB were having almost similar penetration values, the distinction in viscosity value is noticeable whereby control binder has low viscosity value than 7% RWP-MB as displayed in Figure 6.2, hence lower temperature is needed during manufacturing process compared to RWP-modified binder. Binder that had low viscosity value is less viscous, hence the bitumen will begin to show fluidity faster as the temperature increased, then exhibited low softening point value. The domination of polymer additive in the modified binder makes the binder flow differently although having a similar grade of penetration.

Besides that, the modification of binder with polymer (SBS and RWP) has a high viscosity in comparison with base and control binder. This means that the addition of RWP makes the binder stiffer and reduces the workability of binders and thus, may need higher mixing and compaction temperatures during manufacturing. However, stiffer binder at high temperature could improve the resistance of rutting (Amirkhanian et al. 2011).

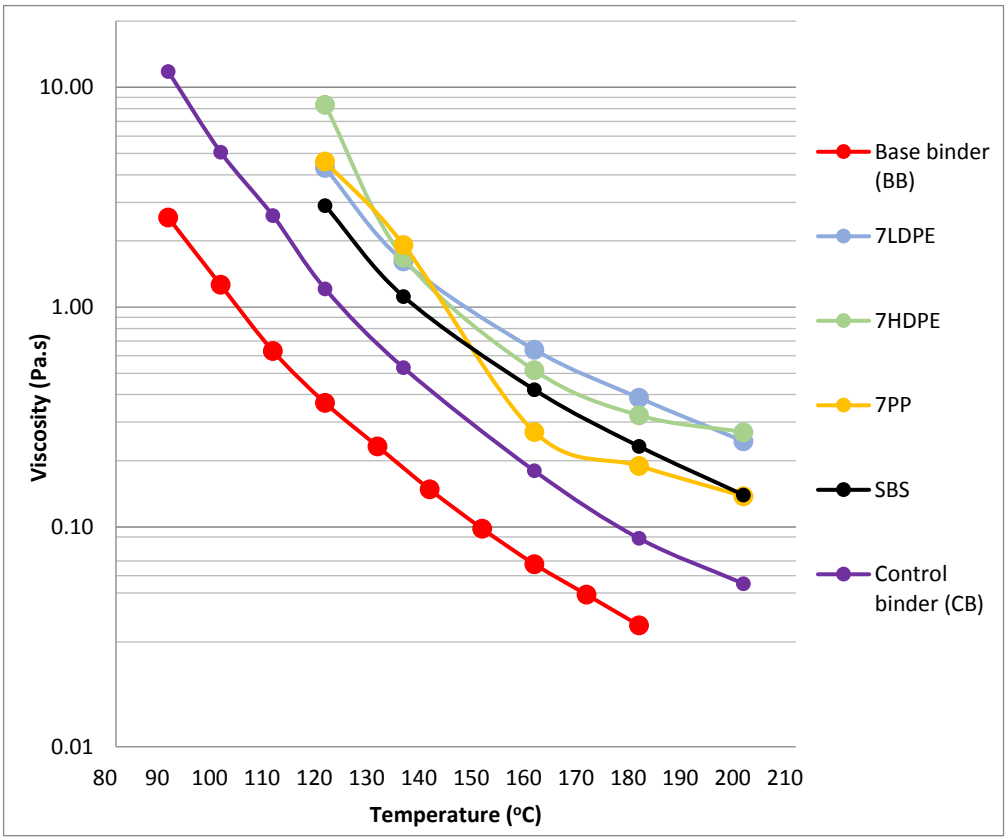


Figure 6.2: Viscosity

6.3 Rheological Properties of Higher Level Modification of RWP-Modified Bitumen

6.3.1 Master Curve

The rheological master curves at a reference temperature of 25°C are produced as shown in Figures 6.4 and 6.5 to evaluate the frequency dependency of complex

modulus and phase angle, respectively of 7% RWP-modified binder, SBS-modified binder, base and control binder.

In comparison with control bitumen, the binder of 7% modification with RWP exhibited uniform shifting towards higher and lower values of complex modulus at low and high frequencies, respectively, regardless of types of RWP added. These different mechanical characteristics are caused by the RWP additive at high and low temperatures which made the binder have lower frequency and temperature susceptibility. Moreover, the fatigue and rutting performance of RWP-modified binder can be expected to be better than control bitumen.

Similar complex modulus curves are demonstrated for 7% modification binders at intermediate and high frequencies for all three types of RWP used and thus, parallel behaviour is expected for all these modified binders at intermediate and low temperatures. However, a significant sequence of complex modulus curves at low frequencies is displayed in Figure 6.3 with the highest complex modulus is reported for 7PP-modified bitumen, followed by 7LDPE-modified bitumen and 7HDPE-modified bitumen.

The SBS-modified bitumen exhibited better performance at low temperature, as evidence by lower complex modulus at high frequencies compared to other binders. But at low frequencies, the SBS and RWP-modified binders displayed higher values of complex modulus. Moreover, both SBS and 7% RWP-modified binders showed the dominance of the polymer network at low frequencies as a result of decrease in slope of the curve. This indicates that the stiffness of the modified binder is improved and hence, better in rutting performance.

The comparison of phase angle master curves as depicted in Figure 6.4 showed that the base bitumen and control bitumen have similar shape of curve at low frequencies but slightly different at high frequencies. Meaning that at high temperatures, both binders are in a viscous state as the phase angle showed a value of 90^{o} towards the end of the curve while at low temperatures, the base binder has viscous behaviour but the control binder tends to exhibit elastic response at this range of frequencies.

Making comparison between 7% RWP-modified bitumen and control bitumen, although having similar penetration grade, binders containing 7% RWP exhibited lower phase angle at low frequencies (Figure 6.4) and the effect of polymer network is clearly observed. The addition of RWP into base bitumen demonstrated improvement in elasticity in comparison with control bitumen and thus, could help the binders to be more rutting resistant by increasing the elastic response of the binder at high temperatures.

As shown for 7% modification bitumen and SBS-modified bitumen, the modification with polymer makes the curves appear similar between them but different magnitude in which the bell shape of phase angle master curve is observed in Figure 6.4 for these binders. At low and high frequencies, both binders exhibited low phase angle with values of 20 to 40^{o} while at intermediate frequencies, the phase angle for these binders is around 60 to 70^{o} and not even approaching 90^{o}. This indicated that the addition of polymer into the binder could significantly increase the elastic response of the binders.

Besides that, there is a jump that causes the appearance of the discontinuous curve particularly for 7PP-modified bitumen. This occurred due to the structure of the

plastomer (Asgharzadeh & Tabatabaee 2013). Polacco et al. (2003) reported that the slipping and breaking can occur in the master curve when close to glass transition (the phase when the polymer transitions from glassy to a fluid condition). The compliance errors associated to the chosen DSR testing geometry may lead to this problem to occur (Airey 2002a). Polacco et al. (2003) suggested the torsion bar geometry could be used to eliminate this problem and give more reliable data.

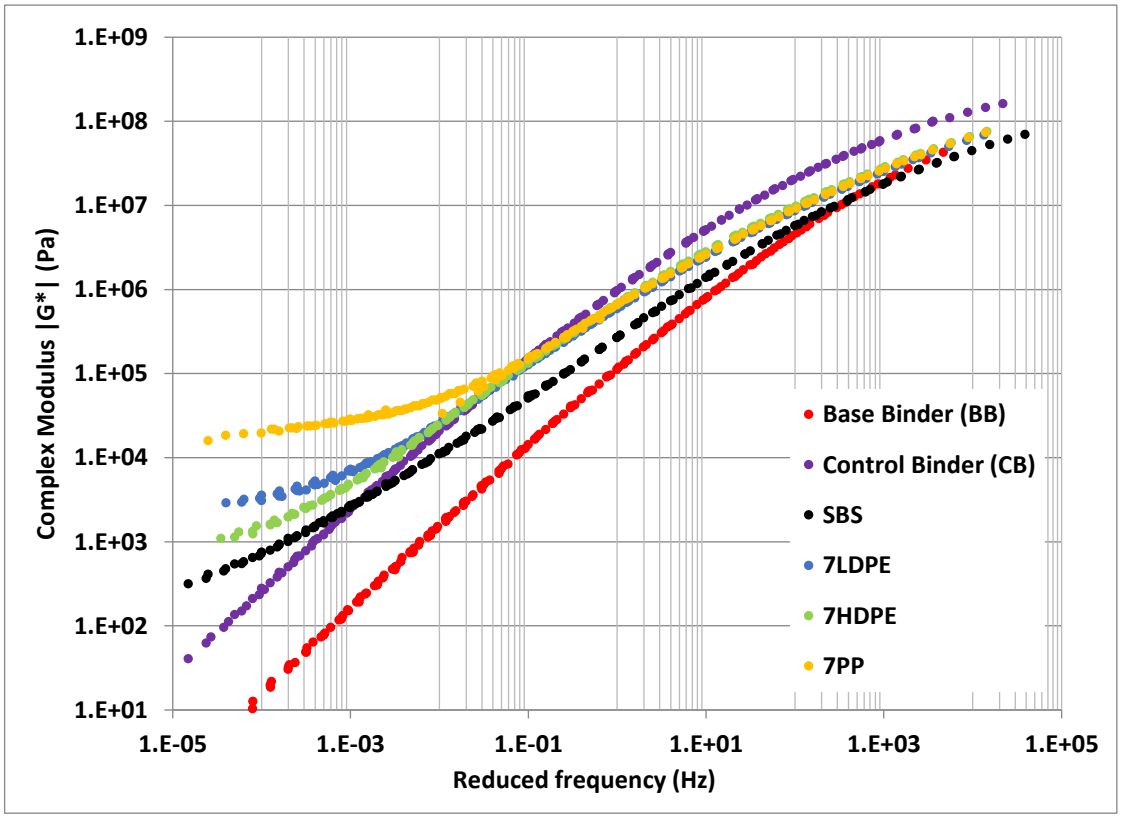


Figure 6.3: Complex modulus master curve for binders at T_f = 25°C for higher level of modification

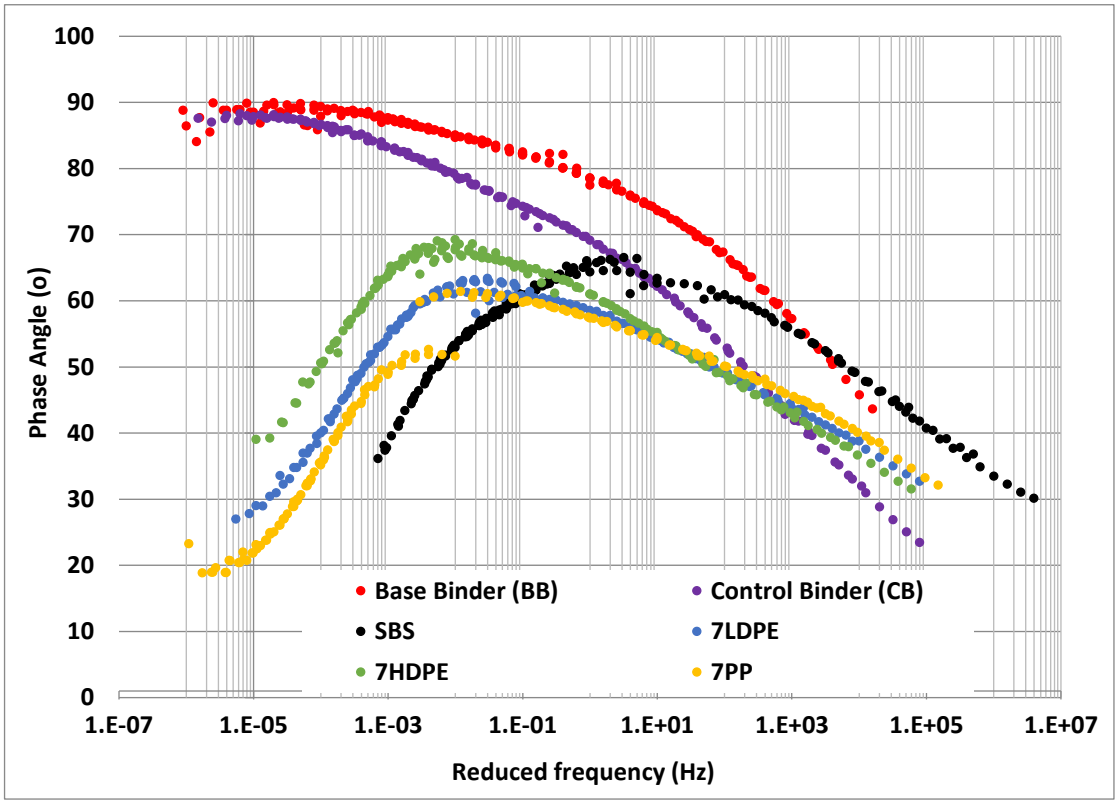


Figure 6.4: Phase angles master curve for binders at T_f = 25°C for higher level of modification

6.3.2 Black Diagram

The Black diagram for higher level modification is displayed in Figure 6.5. It can be observed that at low temperatures, all binders have the same complex modulus and phase angle irrespective of the polymer used and bitumen grade. The curve of Black diagram for base binder and control binder exhibited very similar curves over the range of frequencies and temperatures tested although having different penetration grade. It showed a significant reduction in complex modulus with phase angles shifting towards viscous component at high temperatures and thus, may affect the rutting performance of the asphalt mixture in the field.

Comparison with Black diagram curves of 7% RWP-modified binders and control binder in Figure 6.5 exposes that the modified binders have increased elastic response

and are stiffer at higher temperatures. While at low temperatures, the discrepancy of phase angle and complex modulus between these binders are insignificant. The presence of RWP causes a recovery in elasticity at high temperature and makes the binder harder. Increase in the elastic component at high temperature showed that the behaviour of the binder is dominated by the polymer network as the binder becomes soft (Pérez-Lepe et al. 2005; Jiménez del Barco Carrión 2017). The lower phase angle value with increase in stiffness is favourable as this behaviour will make the modified bitumen more resistance to rutting.

The Black diagram of SBS-modified bitumen and all 7% modified binders illustrated parallel curves as shown in Figure 6.5. As can be seen, at higher temperatures, the elastic responses of modified binders are increased as evidence of decrease in phase angle value coupled with a decrease in complex modulus as frequency decrease.

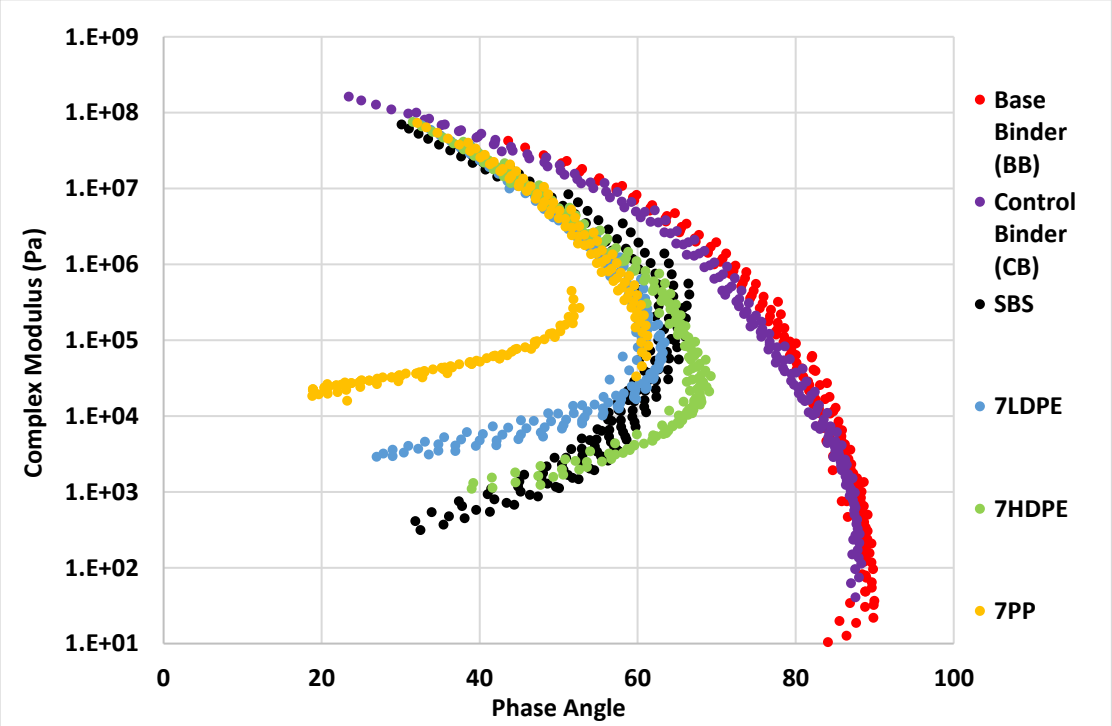


Figure 6.5: Black diagram for higher level modification

6.4 Fatigue

The Linear Amplitude Sweep (LAS) test was conducted to evaluate the resistance to fatigue at test temperatures of 15, 20 and 25^{o}C. All studied binders were aged with RTFO condition before being tested.

6.4.1 LAST test

6.4.1.1 Failure Strain Comparison of Binders

The typical plot of shear stress versus shear strain is used as an indicator for deformation tolerance of the binder (Wang et al. 2017). Figure 6.6, 6.7 and 6.7 presents a plot of shear strain against shear stress obtained from LAS test at tested temperatures of 15, 20 and 25^{o}C, respectively for 7% RWP-modified bitumen, SBS-modified binder, base binder and control binder. During testing, the control bitumen could not be tested at temperature of 15^{o}C because the binder become too stiff and brittle at this temperature and thus, failure occurs when the test just started. As shown in Figure 6.6, the base binder and control bitumen display similar high occurrence of damage irrespective of test temperature as shown by the earliest degradation in the stress responses at strain levels about 15%.

At test temperature of 15^{o}C, the degradation of stress responses can be clearly observed for all modified binders with the 7LDPE-modified bitumen showing the lowest yield stress and highest failure strain among them. As the test temperature increased, the damage occurs for all modified binders (except for 7PP-modified bitumen) but is difficult to identify due to no yield point being observed as the stress response is not significantly decreasing when the applied strain amplitudes increased. This indicates that the all modified binders show their higher elastic response due to the appearance of elastic network formed by the polymer added and thus, become

independent on the applied strain amplitudes. It is hence recommended that the higher strain level namely more than 30% could be used especially when testing polymer-modified bitumen in order to provide a better damage prediction.

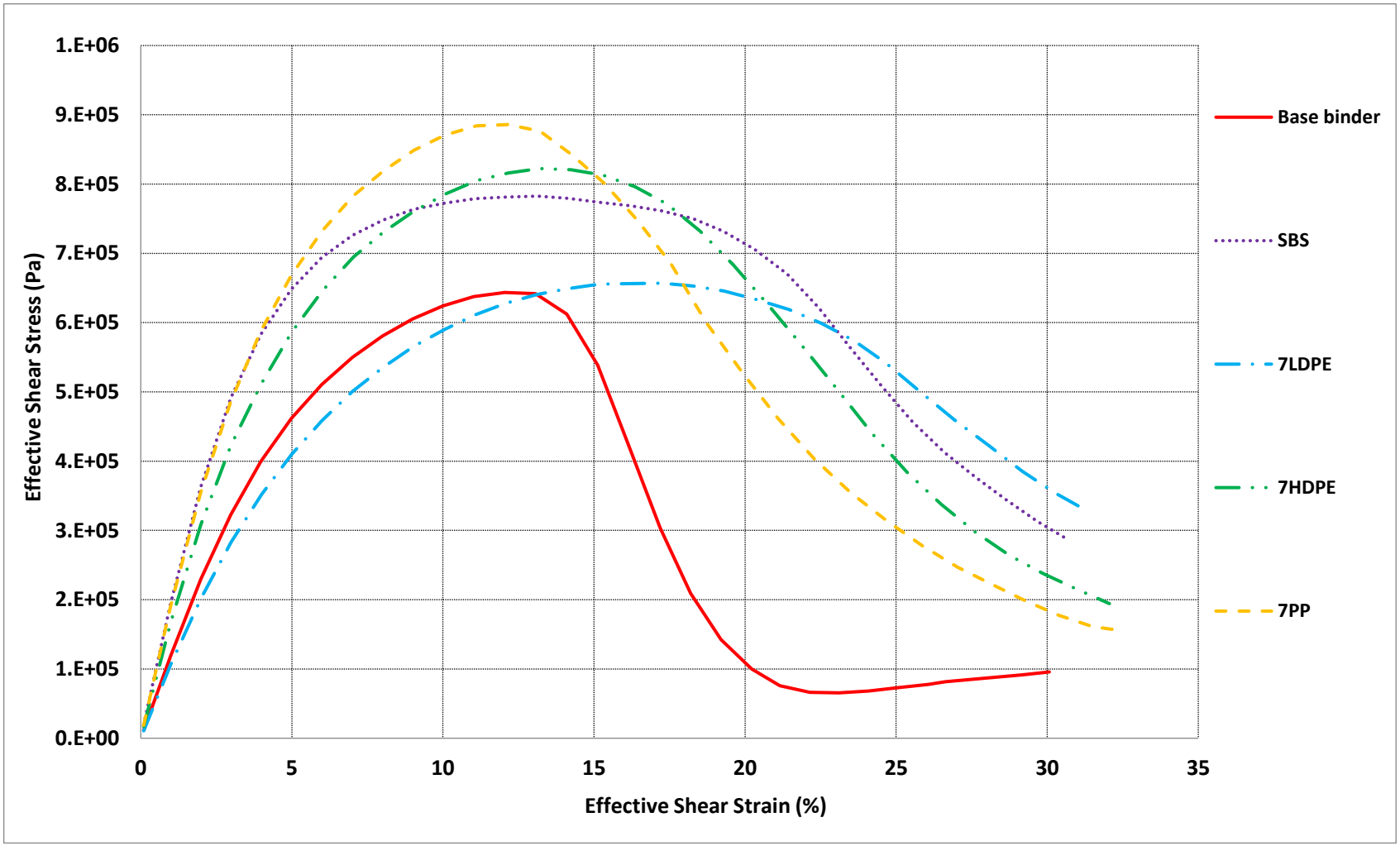


Figure 6.6: Stress versus strain curve from the LAS test for at temperature of 15°C

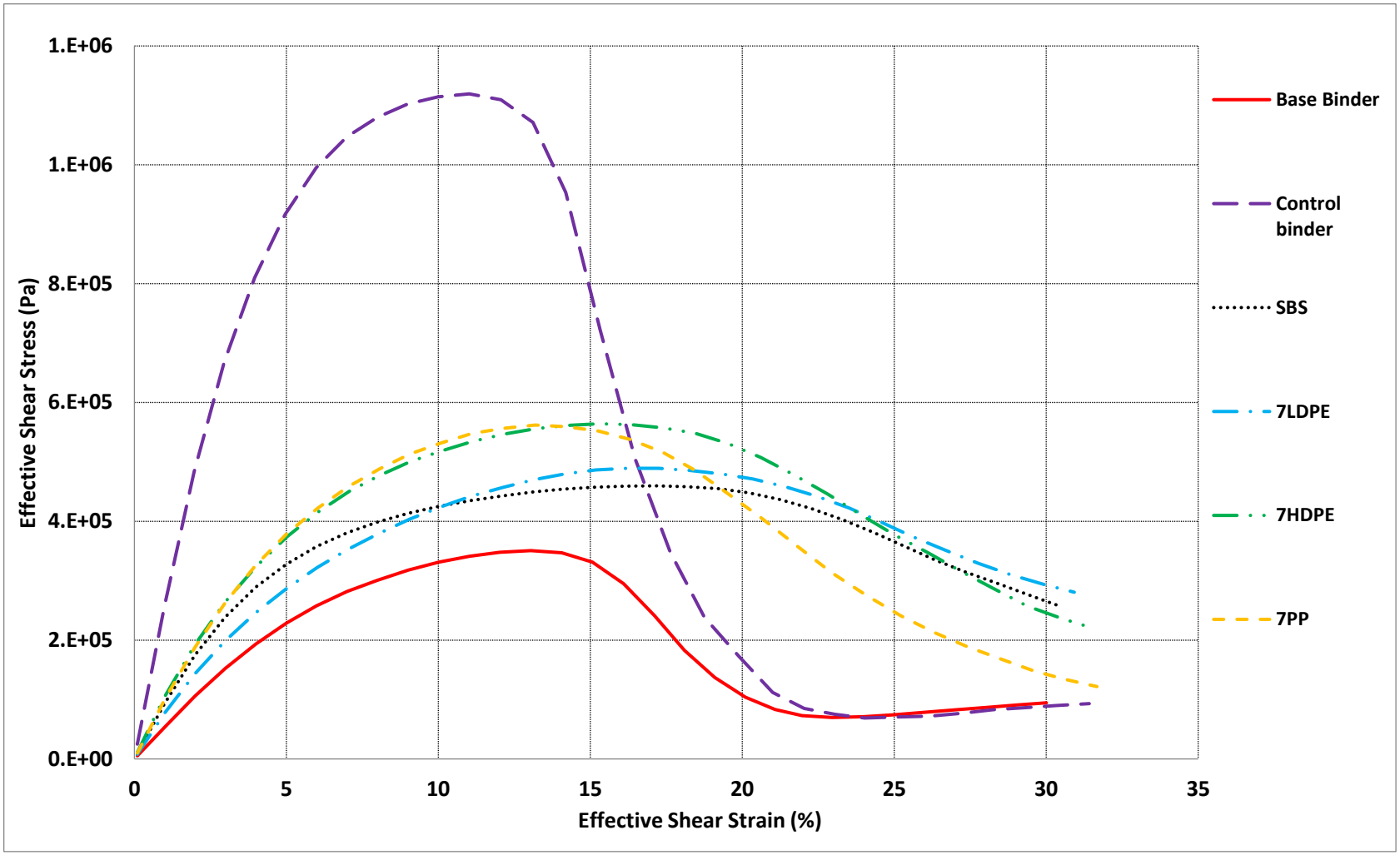


Figure 6.7: Stress versus strain curve from the LAS test for at temperature of 20°C

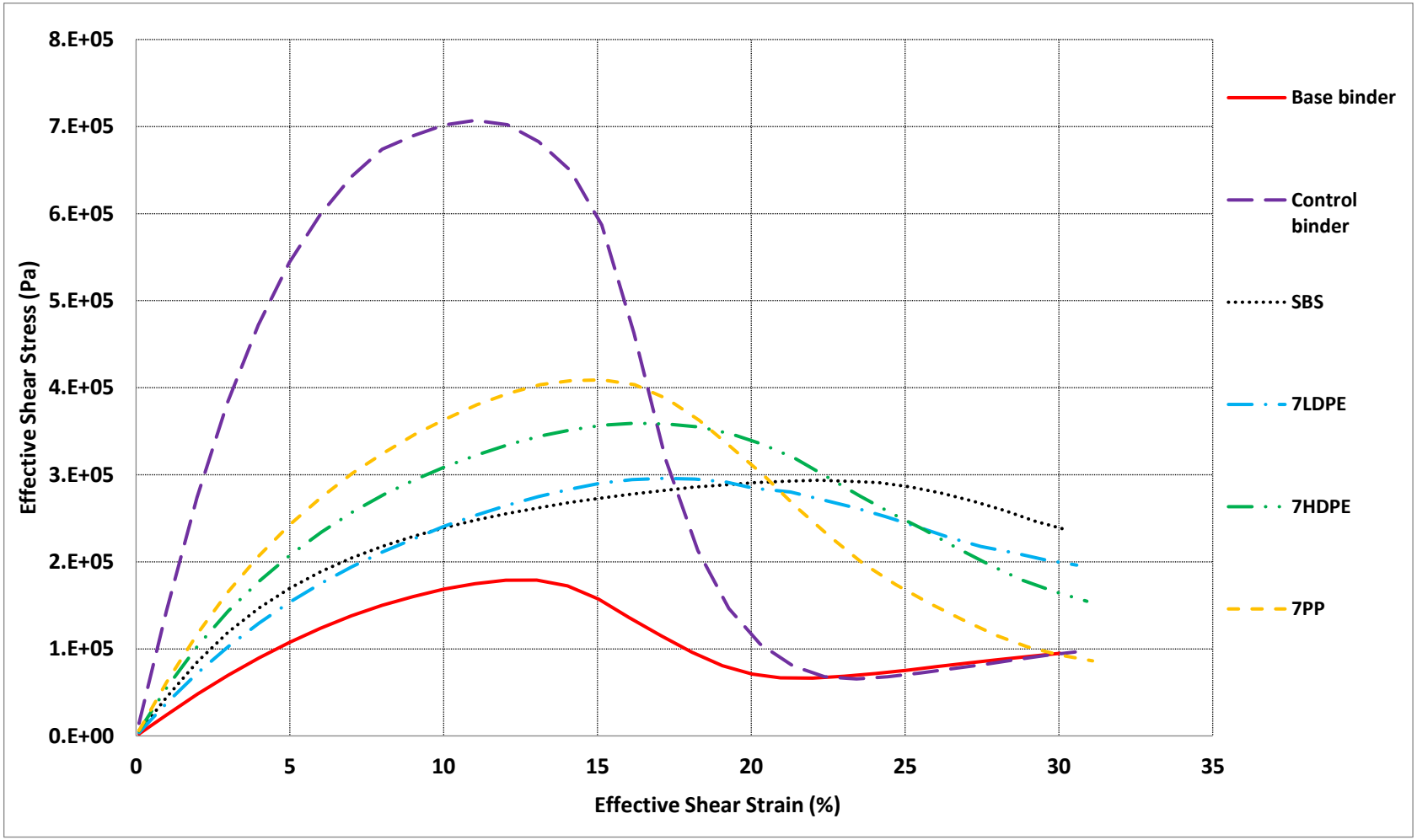


Figure 6.8: Stress versus strain curve from the LAS test for at temperature of 25°C

6.4.1.2 Damage and Failure Analysis

The measured damage characteristics curves for all studied binders are depicted in Figure 6.9, 6.10 and 6.11 at tested temperatures of 15, 20 and 25^{o}C, respectively. Xue et al. (2017) stated that this curve is the relationship between the pseudo stiffness in which refers as the integrity of the material tested as a function of the increasing in damage intensity.

By incorporating 7% RWP into the base binder, the damage characteristics curves are located at higher positions compared to the base bitumen and the control bitumen at all temperatures which indicates that modifying base binder with RWP will improve the ability of the binder to resist more damage. Evaluation among the modified binders displays a similar trend of the damage resistance capability for any test temperature with the 7LDPE-modified bitumen outperforming all these binders followed by the SBS-modified bitumen, 7HDPE-modified bitumen and 7PP-modified bitumen. In addition, the curve of damage characteristic for SBS-modified binder shows a tendency toward a higher position if subjected to a higher strain level. Hintz et al. (2011) stated that testing LAS test at strain levels beyond 20% is required in order to make the binder exhibit more significant damage. The needs for considering higher strain level have been done by Bahia et al. (1999) whereby they stated that it is vital for estimating the performance of the binder in the asphalt mixture.

It can be concluded that the strain levels of the LAS test used in the procedure is not high enough for obtaining damage particularly when the binder could resist the strain applied and remain undamaged.

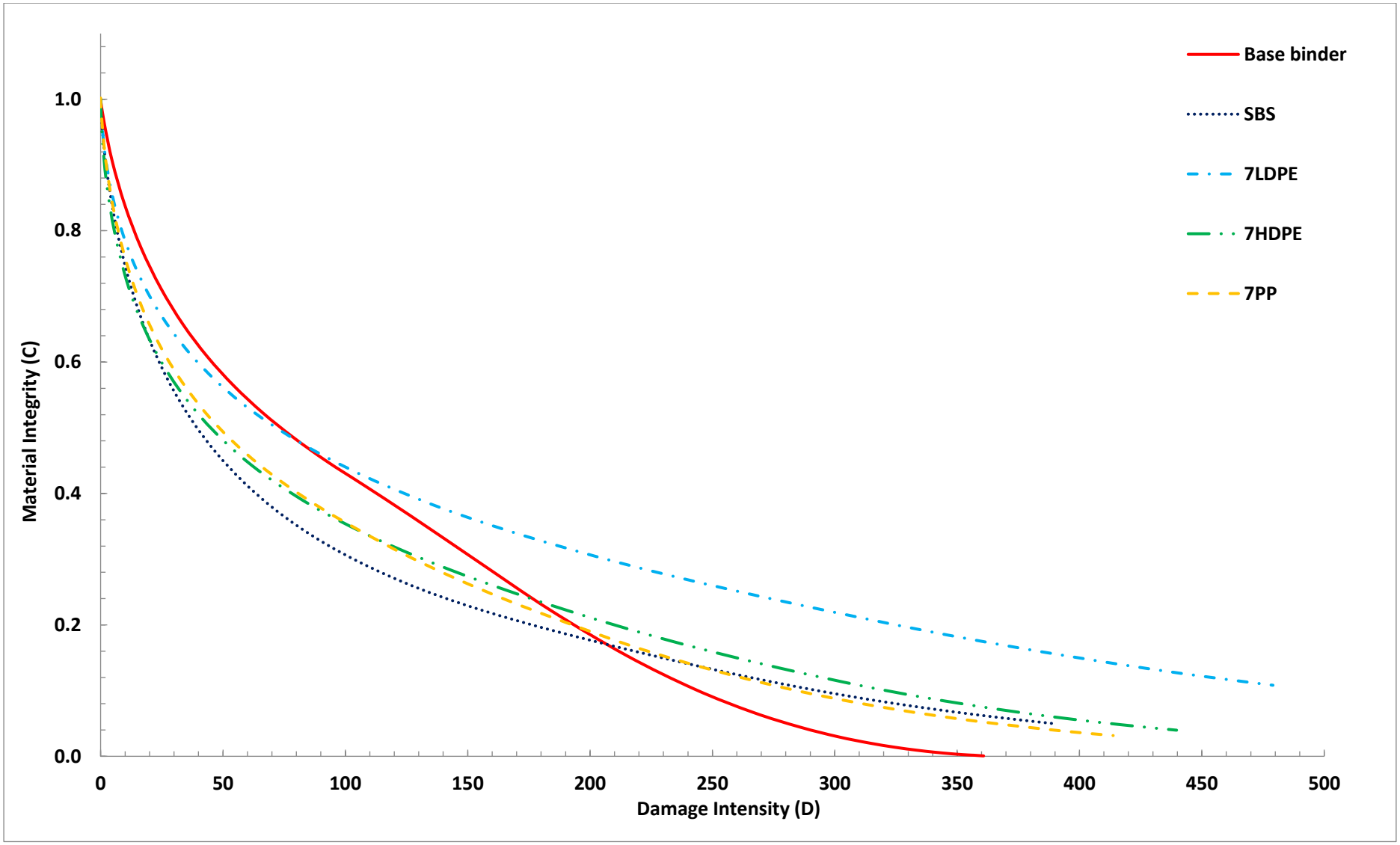


Figure 6.9: Damage characteristics curve for higher level modification binders at tested temperature 15°C

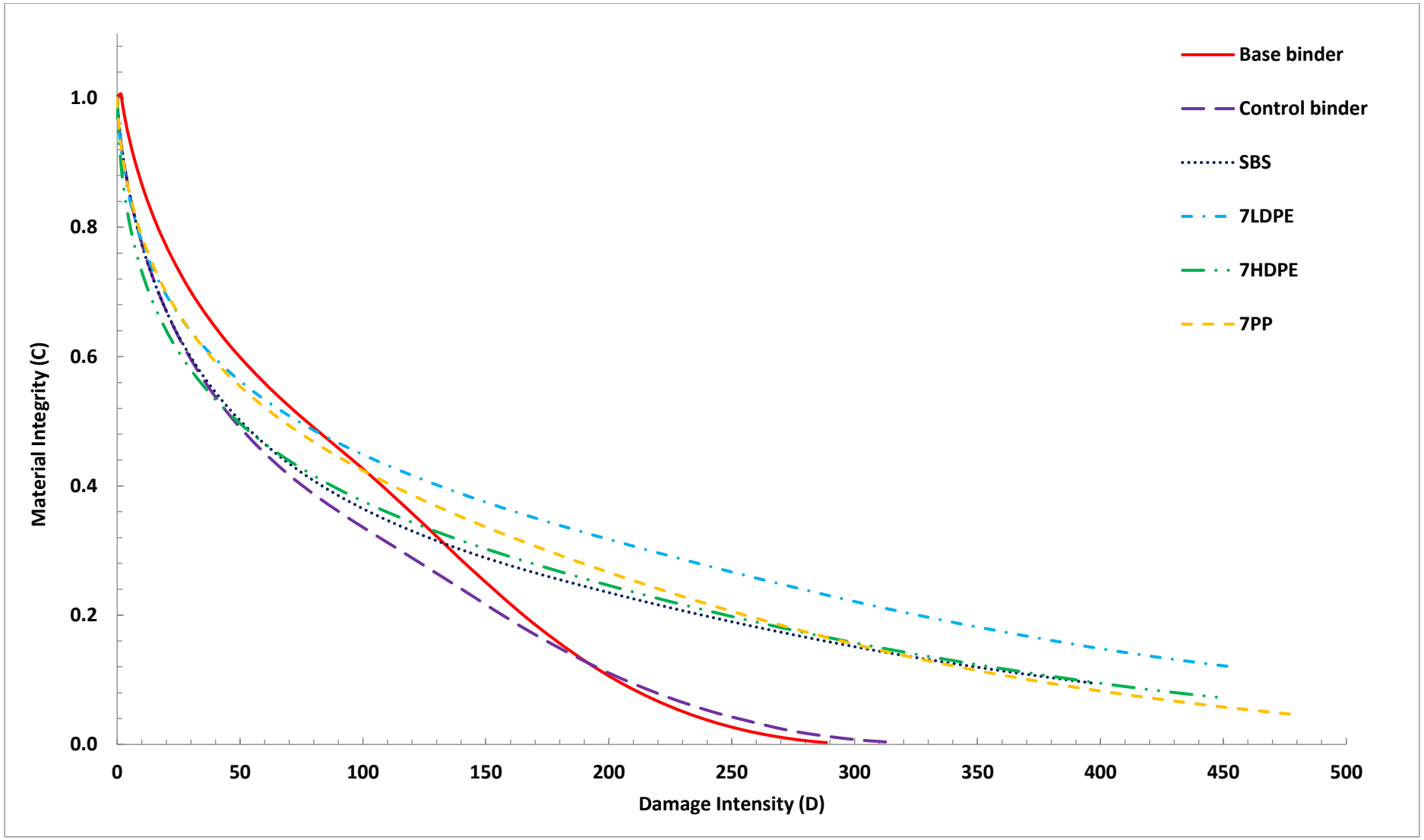


Figure 6.10: Damage characteristics curve for higher level modification binders at tested temperature 20°C

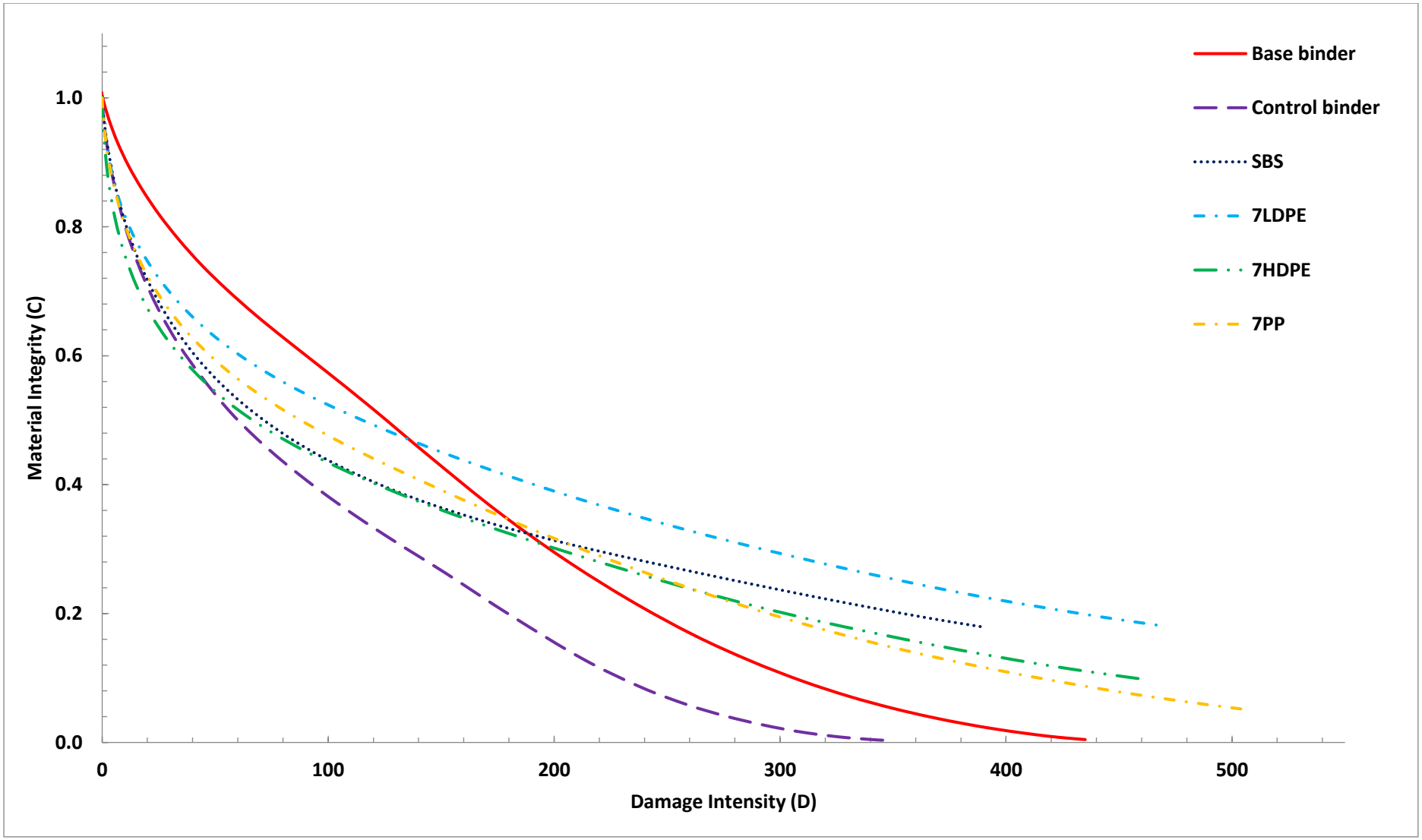


Figure 6.11: Damage characteristics curve for higher level modification binders at tested temperature 25°C

6.4.1.3 Failure Live Prediction of Binders

The log-log plot of fatigue life versus applied strain for 7% RWP-modified bitumen, base binder, control binder and SBS-modified bitumen at three testing temperatures are compared in Figures 6.12, 6.13 and 6.14. The RWP-modified binders display better fatigue performance than base binder at all strain levels irrespective of RWP types used and test temperatures.

Evaluation of the binders with the same penetration grade shows that the fatigue life for the RWP-modified binders is better than control binder. At temperatures of 15 and 25^{o}C, the 7LDPE-modified bitumen had higher fatigue life than other binders at all strain levels. While at 20^{o}C, higher fatigue life is exhibited by the 7HDPE-modified bitumen at low strain levels but later at strain levels higher than 8.5%, the 7LDPE-modified bitumen displayed better fatigue lives. Unfortunately, at testing temperature of 15^{o}C, the LAS test for control binder could not be performed because at this temperature, the binder is too stiff and failure occurs right after the test started as the control binder fails in a brittle state.

As seen in the Figure 6.8 at strain levels more than 9%, the fatigue live of SBS-modified bitumen is more than the RWP-modified binders particularly at temperatures of 20 and 25^{o}C. It is interesting to note that at strain levels below 3.5%, all RWP-modified binders have outperformed the SBS-modified bitumen in term of fatigue performance at all tested temperatures. However, by considering the rate of decrease in fatigue life at all strain levels, the RWP-modified binders have higher decreases in fatigue life than the SBS-modified bitumen as shown by fatigue lines decreasing steeply for RWP-modified binder for all RWP types compared to SBS-

modified bitumen at all temperatures. This indicates that the SBS-modified bitumen is less susceptible to the change of strain level. Moreover, similar behaviour occurs for base binder at higher strain levels. Moghadas Nejad et al. (2017) reported that the effect on fatigue improvement is less at higher strain levels for most of the modifiers due to the polymer network tending to break up when the strain and stress level increases.

Figures 6.15 and 6.16 show the predicted fatigue lives of the base binder, control binder, RWP-modified binders and SBS-modified bitumen at strain levels of 2.5 and 5%, respectively. The range bar displayed in the figures denoted the maximum and minimum values for the replicates. The strain level of 2.5% denoted the binder used in strong pavements with asphalt layers thicker than 114.3 mm (4.5 inches) and 5% strain represents the binder strain used for thinner asphalt layers (weak pavement) (Bahia et al. 2013; Moghadas Nejad et al. 2017). As illustrated in both figures, adding RWP as modifier at higher level modification could improve the fatigue life of the base binder. It can be observed that the RWP-modified binders displayed better fatigue live than SBS-modified bitumen at strain levels of 2.5% and 5%. Therefore, the effect in improving fatigue performance is pronounced in thick and thin asphalt pavements for the RWP-modified binders.

Moreover, no specific trend of fatigue life variation could be observed as the temperature increased for the RWP-modified binders. However, base binder exhibited least fatigue resistance at both strain levels for all testing temperatures as shown by lowest fatigue lives. Meanwhile for SBS-modified bitumen, when the test temperature increases, insignificant reduction in fatigue life is displayed at strain

level of 2.5% but the opposite way has occurred for 5% of strain level, fatigue life is increases indicates this bitumen could resist fatigue cracking better than other binders. Generally, increased in temperature would decrease the fatigue lives for all the binders and indicated lower temperature susceptibility except for SBS-modified bitumen at 5% strain level. For both strain levels, an evaluation of RWP-modified binders show that the fatigue lives of 7LDPE-modified bitumen are higher than other binders at testing temperatures of 15 and 25°C while 7HDPE exhibited highest fatigue performance at temperature of 20°C.

Based on the fatigue lives of the five investigated binders at test temperature of 20°C and strain levels of 2.5 and 5%, the LAS ranking is as follow:

7HDPE > 7LDPE > 7PP > SBS-modified binder > control binder > base binder

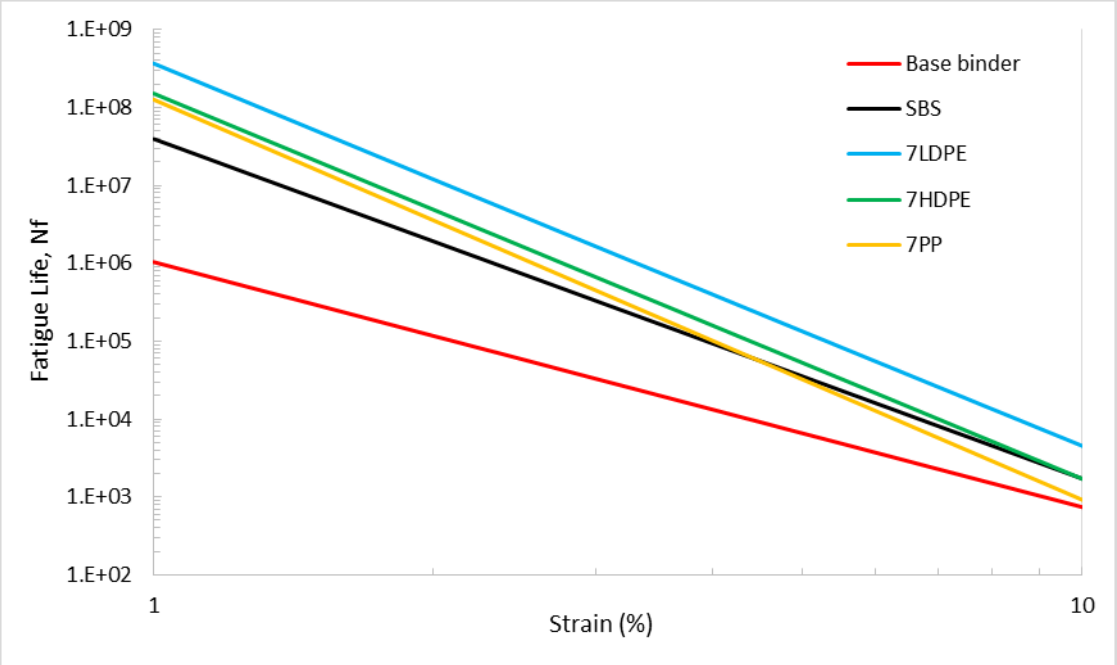


Figure 6.12: Fatigue life against strain levels at temperature of 15°C for 7% modification level, control binder and SBS-modified bitumen

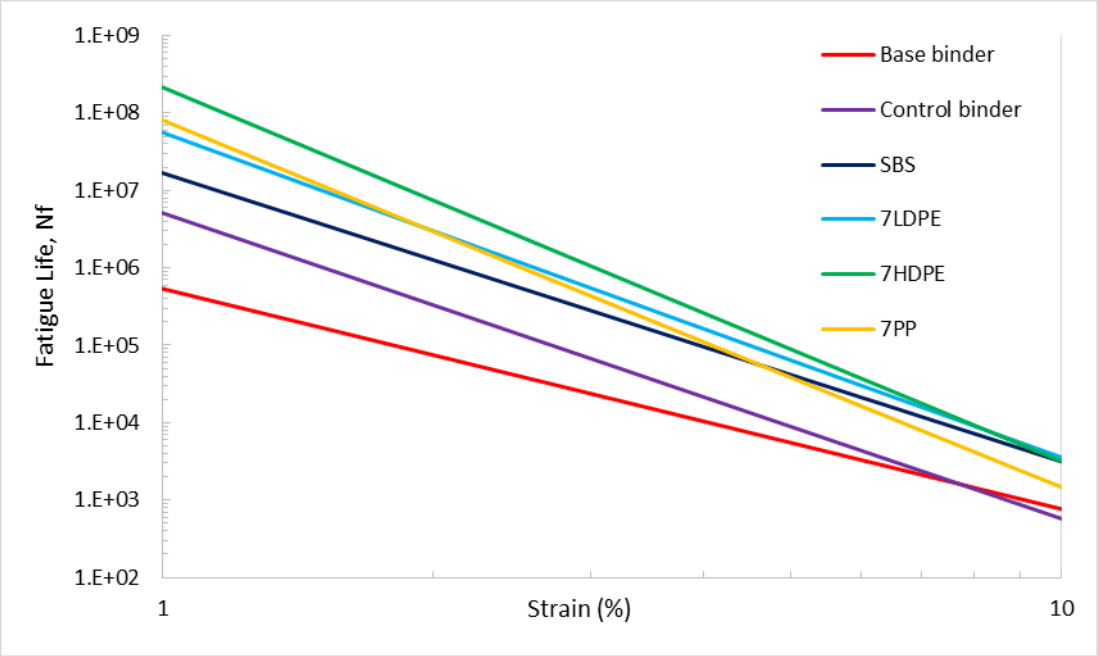


Figure 6.13: Fatigue life against strain levels at temperature of 20°C for 7% modification level, control binder and SBS-modified bitumen

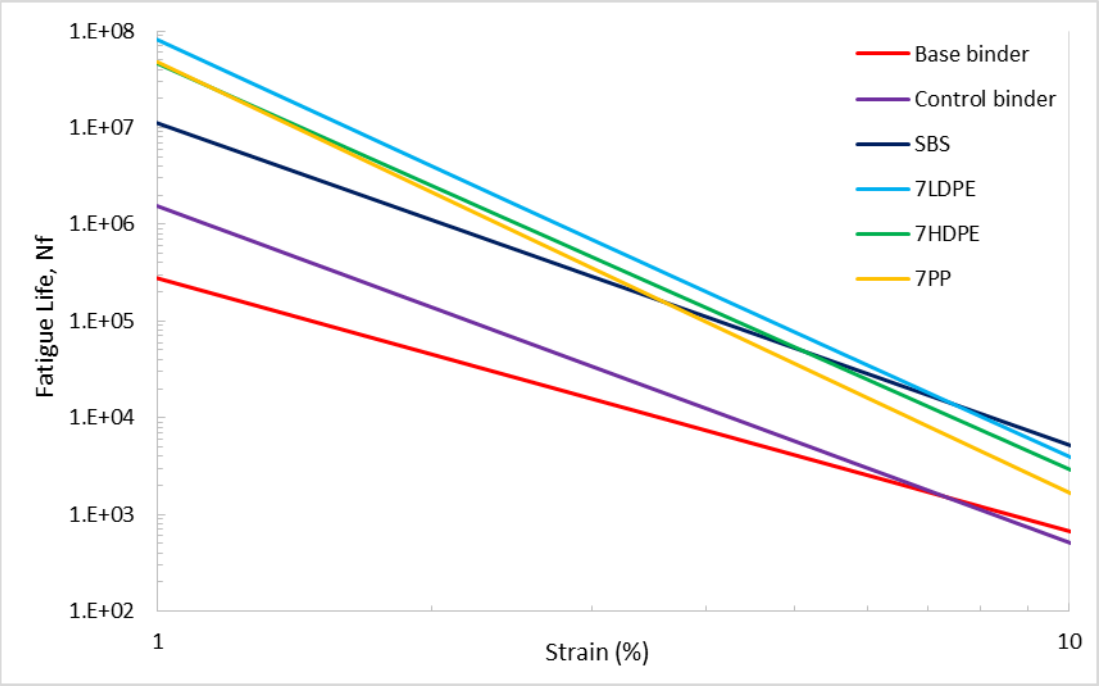
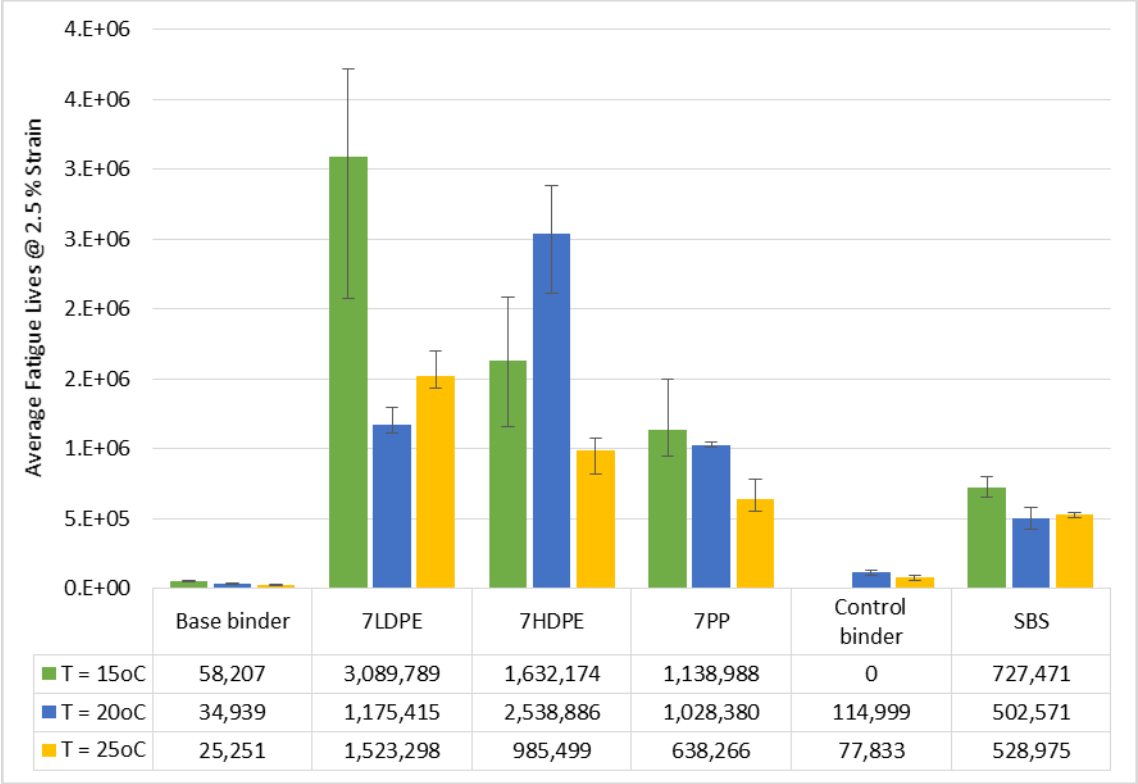
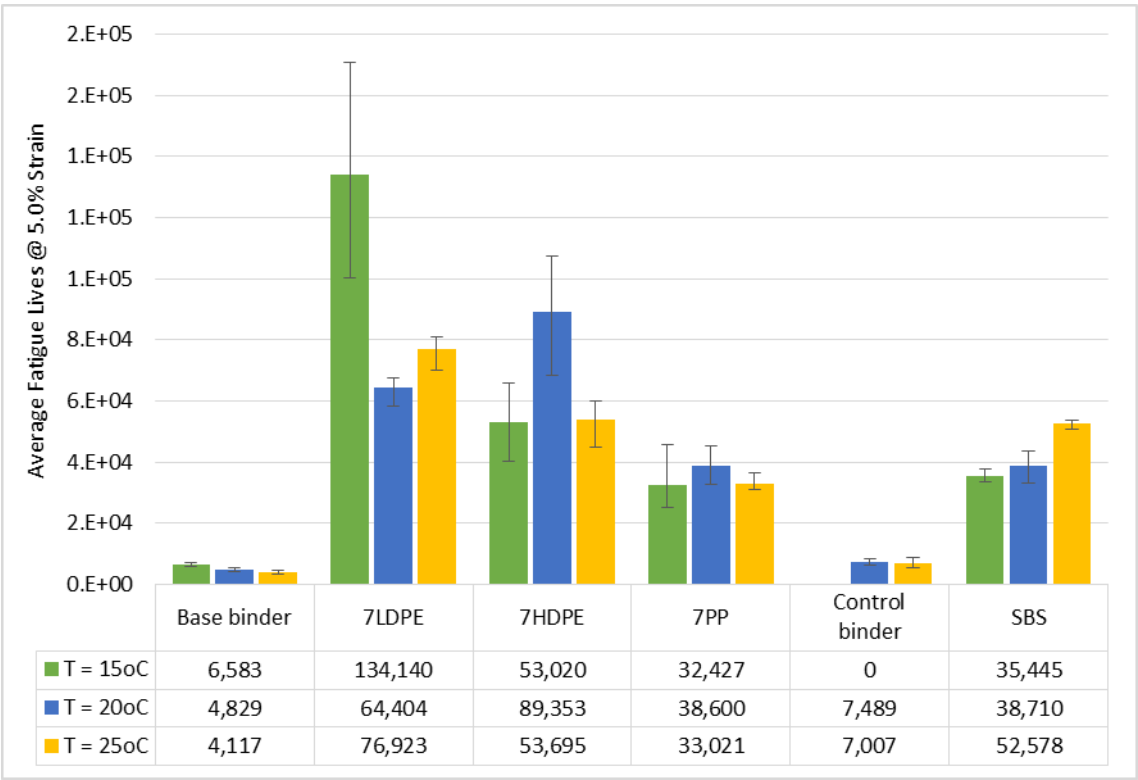


Figure 6.14: Fatigue life against strain levels at temperature of 25°C for 7% modification level, control binder and SBS-modified bitumen



	Base binder	7LDPE	7HDPE	7PP	Control binder	SBS
T = 15oC	58,207	3,089,789	1,632,174	1,138,988	0	727,471
T = 20oC	34,939	1,175,415	2,538,886	1,028,380	114,999	502,571
T = 25oC	25,251	1,523,298	985,499	638,266	77,833	528,975

Figure 6.15: Variation of fatigue life with temperature of 15, 20 and 25^{O}C at strain level of 2.5%



	Base binder	7LDPE	7HDPE	7PP	Control binder	SBS
T = 15oC	6,583	134,140	53,020	32,427	0	35,445
T = 20oC	4,829	64,404	89,353	38,600	7,489	38,710
T = 25oC	4,117	76,923	53,695	33,021	7,007	52,578

Figure 6.16: Variation of fatigue life with temperature of 15, 20 and 25^{O}C at strain level of 5%

6.4.2 DENT test

For this test, the samples tested have been aged using RTFOT and PAV to simulate the long term ageing due to the fact that fatigue appears during service life of the pavement. The test was done at test temperatures of 10 and 20^{o}.

6.4.2.1 Specific Total Work of Failure

The assumptions that underlie the essential work of fracture method (as described in Section 3.4.2) have been verify in Section of 5.5.2.1 which states that the DENT test is valid, and the specific total work of fracture analysis could be used in this study. Hence, Figure 6.17 represents the comparison of specific total work of fracture for base binder blended with 7% RWP-modified binders, base binder, control binder and SBS at temperatures of 10 and 20^{o}C. The lines in Figure 6.17 showed that the energy analysis according to the formula of specific total work of fracture is valid as evidenced by straight lines displayed R^2 values more than 0.95 (high accuracy) for all binders. The results for control binder at 10^{o}C could not be determined because this binder tends to fracture early without yielding or in other word, failure occurs in brittle state and thus, unable to fulfil the ductile failure state (Jafari & Babazadeh 2016). Thus, no data is recorded for control binder at 10^{o}C.

It can be seen that SBS-modified bitumen had higher values of specific work of fracture than 7% RWP-modified binders at both temperatures while base binder had lowest specific total work of fracture among all the binders tested, indicating that more energy is required to fracture the SBS-modified bitumen and thus it has more fracture resistance. Making comparison with 7% RWP-modified binders, at testing temperature of 20^{o}C, the control binder exhibits small increase in specific work of fracture compared to the RWP-modified binders while as the temperature decrease

to 10^{o}C, the specific work of fracture of control binder could not be recorded as the sample is too brittle at temperature of 10^{o}C. Thus, the yielding capacity of 7% RWP-modified binders may be considered better than the control binder at low temperatures. Moreover, a specific trend is observed among the 7% RWP-modified binders in which the 7LDPE-modified binder had more specific total work of fracture followed by the 7HDPE and lastly, 7PP-modified binder at both temperatures.

The slope of the specific work of fracture line in Figure 6.17 indicates the plastic work of fracture (denoted as βw_p) of the binders which represents the energy dissipated in ductility, plasticity and tearing (Jafari & Babazadeh 2016). At test temperature of 10^{o}C, the slope of the line for modified binders namely SBS, 7LDPE, 7HDPE, 7PP was measured to be 4.65, 1.89, 1.47 and 1.18 kJ/m^2, respectively while the lowest slope is exhibited by the base binder with 0.44 kJ/m^2. As the test temperature increase up to 20^{o}C, the slope of the modified binders namely SBS, 7LDPE, 7HDPE, 7PP was measured to be 1.58, 0.86, 0.47 and 0.38 kJ/m^2 while for control and base binder was found to be 0.67 and 0.03 kJ/m^2. The SBS-modified bitumen displays higher values of βw_p among other binders at both of testing temperatures while the base binder had smaller βw_pvalues than the RWP-modified binders. The lower βw_pvalue may make the binder more susceptible to fatigue resistance (Bahia et al. 2001). Moreover, the reduction of plastic work of fracture for all binders is more than 50% when the testing temperature is increased from 10 to 20^{o}C except for the control binder. This showed that the energy needed for performing plastic deformation outside the fracture zone decreases as the temperature increases.

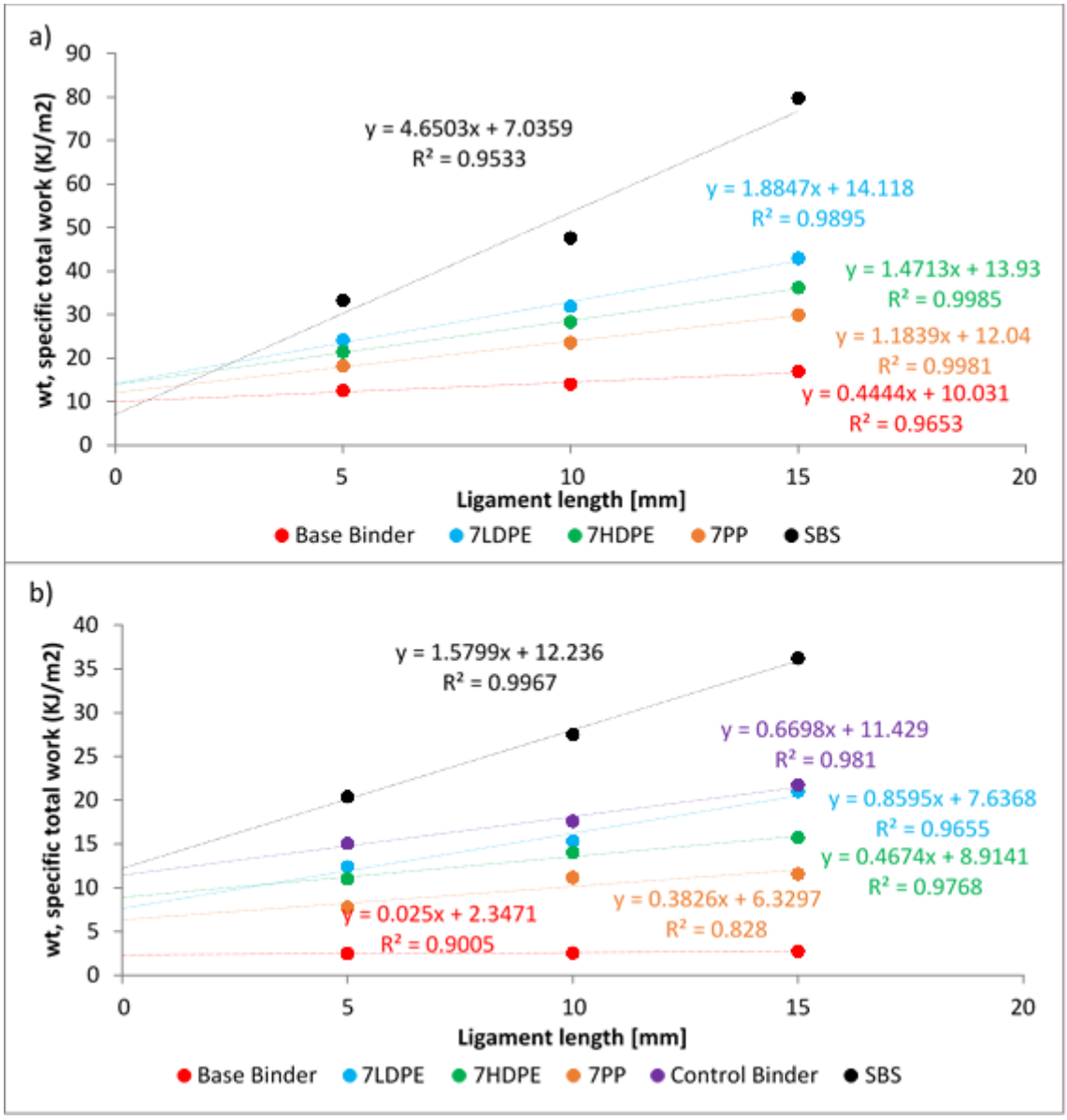


Figure 6.17: Specific total work of fracture for higher level modification at testing temperature of a) 10°C and b) 20°C

6.4.2.2 The Essential Work of Fracture and CTOD

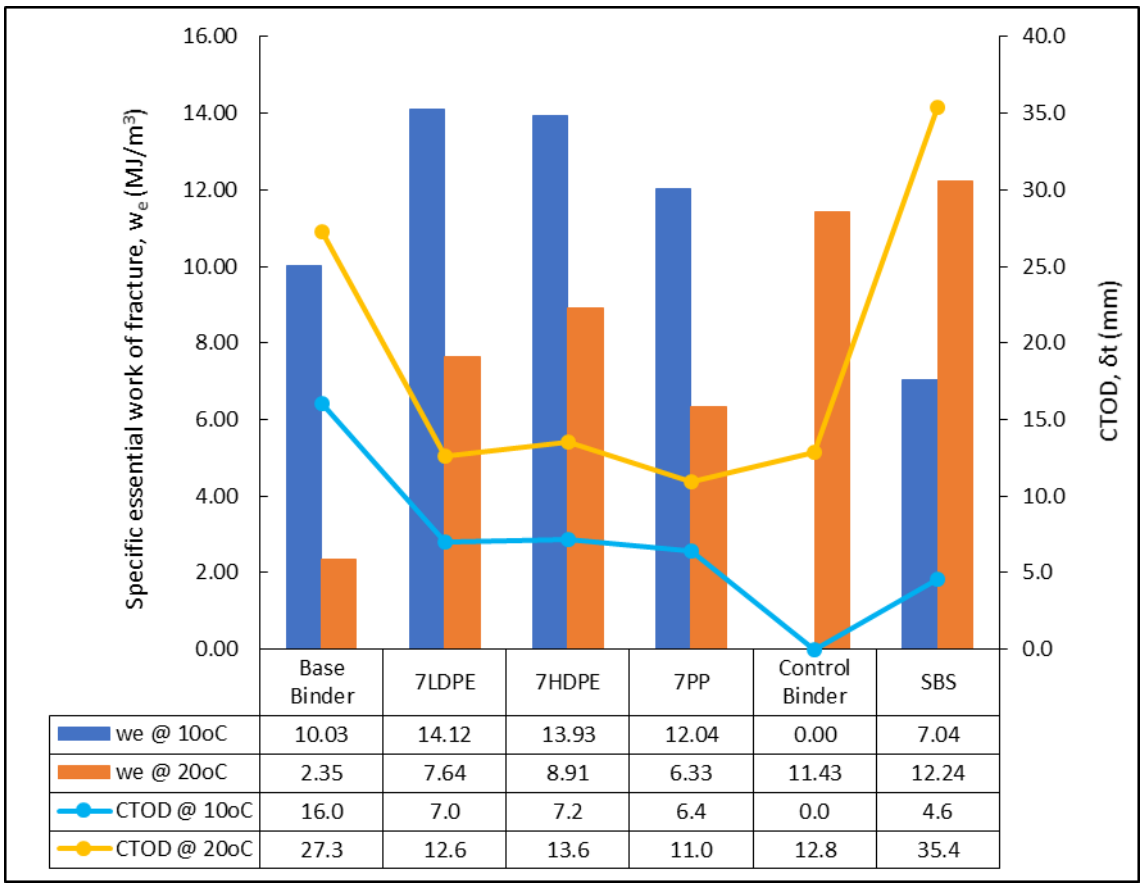
Figure 6.18 provides the comparison of the essential work of fracture (w_e) and CTOD value between 7% RWP-modified binders, base binder, control binder and SBS-modified bitumen. The data of both parameters is missing at test temperature of 10°C for the control binder as the binder tends to fracture at the brittle state in which the fast crack propagation occurred before yielding of the ligament section (Martinez

et al. 2009). Generally, the criterion of CTOD is related to the crack opening before the extension and can be considered as the capability of plastic flow of the polymer (Bárány et al. 2010) in which it measures the strain tolerance in the appearance of significant constrain in the ductile state (Subramani 2009). Higher values of w_e and CTOD are desired for better fatigue resistance and tolerance to strain (Andriescu et al. 2004; Singh et al. 2017).

In comparison with the base binder, the addition of 7% RWP regardless of RWP type into base binder improved the w_e values but there is a reduction on the CTOD values, showing a poorer resistance to fracture. The higher energy is required to fracture the sample means that the sample is stiff and less elongate. This indicates that the RWP-modified binders are not flexible due to the limited improvement in the elasticity (Zhu et al. 2014).

The comparison of the control binder and base binder blended with the 7% RWP revealed that the RWP-modified binders have lower values of w_e and CTOD at test temperature of 20^{o}C. As the temperature decrease to 10^{o}C, the fracture properties of control binder could not be measured and thus, RWP-modified binders may have better fracture resistance at low temperatures. While the SBS-modified binder exhibited interesting fracture behaviour which at 10^{o}C, the w_e and CTOD values are lowest and as the testing temperature increase to 20^{o}C, these parameters increases from 7.04 to 12.24 kJ/m^2 and from 4.6 to 35.4 mm for the w_e and CTOD values, respectively and was found to be the highest among other binders. This indicates that the SBS-modified binders are more resist to fracture at high temperatures.

It can be seen that the 7% RWP-modified binders are less susceptible to fracture in comparison to the control binder particularly at low temperatures however it seems more prone to fracture than the base binder at both testing temperatures. Moreover, a clear ranking can be observed among RWP-modified binder at both testing temperatures based on the CTOD value and at both testing temperatures, the 7HDPE-modified bitumen has higher strain tolerance followed by 7LDPE-modified bitumen with 7PP-modified bitumen having the worst fracture resistance under the DENT test.



	Base Binder	7LDPE	7HDPE	7PP	Control Binder	SBS
we @ 10oC	10.03	14.12	13.93	12.04	0.00	7.04
we @ 20oC	2.35	7.64	8.91	6.33	11.43	12.24
CTOD @ 10oC	16.0	7.0	7.2	6.4	0.0	4.6
CTOD @ 20oC	27.3	12.6	13.6	11.0	12.8	35.4

Figure 6.18: The essential work of fracture and CTOD values for higher level modification at testing temperature of 10°C and 20°C

6.5 Rutting

The Multiple Stress Creep Recovery (MSCR) test was conducted to evaluate the rutting resistance at test temperatures of 40, 60 and 70°C. All studied binders were aged starting with RTFO and then PAV condition before being tested.

6.5.1 MSCR Test

6.5.1.1 Non-recoverable Compliance (J_{nr}) and Percent of Recovery ($\%R$)

Table 6.3 presents the results of the non-recoverable creep compliance (J_{nr}) values and percentage of recovery ($\%R$) at three stress level namely 0.1, 3.2 and 12.8 kPa for all the selected temperatures. A binder that had a higher rut sensitivity would exhibit higher values of J_{nr} and thus, a lower value of J_{nr} is preferred for a binder to be rut resistant (Saboo & Kumar 2016). As shown, the base binder has higher J_{nr} compared to other binders at all stress level for all temperatures and thus, highly prone to rutting. The inclusion of 7% of RWP into the base binder resulted in significant improvement in the rutting resistance after the repetition of loads as shown by the lower value of J_{nr} for all stress levels and temperatures.

Comparison between RWP-modified binders with the control binder having similar penetration grade displays that the J_{nr} values for these binders still remain within the limit (maximum J_{nr} values is 4.5 kPa^{-1} for standard traffic) when the stress level increase up to 12.8 kPa at testing temperatures of 40 and 60°C. Particularly, at these two temperatures, the RWP-modified binders have J_{nr} values below 0.5 kPa^{-1} at stress level of 3.2 kPa in which according to AASHTO, these modified binders could be used in the extremely heavy traffic condition. When the test temperature increases up to 70°C, the control binder and 7% RWP-modified binders regardless of

RWP used exhibited J_{nr} values within the limit up to the stress applied of 3.2 kPa but as the stress level increased to 12.8 kPa, the J_{nr} values are higher than the limit. This means that these binders may have an acceptable rutting performance at road pavement temperatures up to 70^{o}C and loading conditions below 12.8 kPa. However, the control binder had higher J_{nr} values compared to the 7% RWP-modified binders and thus, it can be concluded that the 7% RWP-modified binders may perform better in rutting performance.

The SBS-modified binder displays superior rutting resistance as shown by lower J_{nr} values compared to the other binders at higher stress level for all temperatures. Although the J_{nr} values for SBS-modified binder are higher than the RWP-modified binders at a stress level of 0.1 kPa, these values become lower than the RWP-modified binders when the temperature and stress applied is increased which indicates that the SBS-modified binder has higher rutting resistance. According to the J_{nr} values in Table 6.3, at temperatures of 60 and 70^{o}C, the SBS-modified binder may be suitable to be used under very heavy traffic conditions as evidenced by the J_{nr} values being lower than 0.5 kPa^{-1} at these temperatures. The RWP-modified binders may have similar conditions with SBS-modified binder at a temperature of 60^{o}C while at a temperature of 70^{o}C, the RWP-modified binder is only suitable for the heavy traffic condition.

It can be seen that as the temperature and stress level increased, a significant growth is observed for the J_{nr} value. Based on J_{nr} values at a temperature of 40^{o}C, in terms of its rutting performance, the overall ranking of the six binders as follows:

7HDPE > 7PP > SBS > 7LDPE > Control Binder > Base Binder

Table 6.3: J_{nr} of higher level modification at all temperatures

Binder	Jnr (kPa^{-1})								
	40°C			60°C			70°C		
	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa
Base Binder	0.2883	0.2986	0.3324	6.6177	7.6675	13.0414	23.8200	25.0556	-
Control Binder	0.0247	0.0245	0.0254	0.9089	0.9817	1.1426	4.0100	4.4584	5.9295
SBS	0.0085	0.0083	0.0082	0.0328	0.0250	0.1000	0.0926	0.1000	0.6670
7LDPE	0.0021	0.0132	0.0184	0.0067	0.3359	0.7278	0.2019	1.3407	4.6057
7HDPE	0.0003	0.00033	0.0031	0.0057	0.4036	0.9055	0.0746	1.1181	10.0734
7PP	0.0002	0.00031	0.0097	0.0008	0.4078	1.0276	0.0004	0.8453	6.9480

The percentage of recovery ($\%R$) values of the higher level of modification binders, the base and control binder and the SBS-modified binder for the test temperatures and stress levels considered is shown in Table 6.4. In order to reduce rutting, higher $\%R$ value is vital which indicates that the binder can recover more amounts of deformation when subjected to the repetition of loads (Hossain et al. 2015a; Jafari & Babazadeh 2016).

The addition of RWP into base binder improved the recovery response of the binder significantly and at the same time showed higher recovery value in comparison with the control binder for all testing temperatures. Besides that, some of the result shows a negative value of recovery particularly at higher level of stress and temperature. The combination of high stress level with low modification level

and tested at high temperatures are the cause of this situation occurring because the reliable results are difficult to obtain at very high temperature. However, the 7HDPE-modified binder had negative recovery values even though modified at a high level of modification. The negative value of recovery percentage was generated from the strain curve that increases even in small amounts during the unloading phase due to the inability of certain rheometer to unload the stress applied as fast as demanded (Hossain et al. 2015a; Jafari & Babazadeh 2016).

When it comes to the SBS-modified binder and the RWP-modified binders, it shows that these two binders have almost similar $\%R$ with values more than 90% particularly at a stress level of 0.1 kPa for temperatures of 40 and 60^{o}C. When the stress level and temperature increased, the SBS-modified binder maintained its higher recovery response while the 7% RWP-modified binders demonstrate a significant reduction in $\%R$ values. However, at temperature of 40^{o}C, the $\%R$ of SBS-modified binder displays insignificant increase as the stress level increased and shows that the elastic response of the SBS-modified binder is not dependent on the stress applied, meaning that this binder is able to withstand all loading conditions when the road temperature is around 40^{o}C due to the elastomer content of SBS. Among the three RWP types used, the PP exhibits higher percentage of recovery at a stress level of 0.1 kPa for all tested temperatures but as the applied stress and temperature increases, the recovery values for PP-modified binder decreases and become lowest among other RWPs which indicates that the PP-modified binder may not suitable to be used in unexpected conditions of the temperature and load.

Overall, a general trend can be observed for all binders in which the higher the testing temperature and stress applied, the higher the J_{nr} values and the lower the percentage of recovery.

Table 6.4: R values of higher level modification at all temperatures

Binder	Recovery, R (%)								
	40°C			60°C			70°C		
	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa	0.1 kPa	3.2 kPa	12.8 kPa
Base Binder	15.5	12.3	5.0	8.5	0.0	-1.1	0.9	-1.3	-
Control Binder	39.4	38.1	35.1	0.9	1.0	1.1	4.0	4.5	5.9
SBS	92.2	92.4	92.7	96.2	97.2	88.0	95.3	90.0	63.4
7LDPE	92.1	60.3	47.8	95.1	28.5	9.2	61.0	14.8	1.4
7HDPE	94.0	94.6	71.9	93.9	13.3	3.1	57.6	8.6	-0.1
7PP	98.0	96.8	51.1	98.6	10.7	2.5	97.3	4.3	0.2

6.5.1.2 Stress Sensitivities of Binders

D'Angelo (2009a) stated that the J_{nr} could be used to evaluate the dependence of the binder on stress at certain temperatures as this parameter is more sensitive to the stress applied. Figures 6.19, 6.20 and 6.21 depicted a plot of the non-recoverable compliance (J_{nr}) of the base and control binder, 7% RWP-modified binders and SBS-modified binder over different stress levels at testing temperatures of 40, 60 and 70°C, respectively. All binders have been tested up to a stress level of 28.6 kPa to see the variation of the stress sensitivity of the binder at a higher stress level.

As can be seen in all figures, the J_{nr} value of the base binder and control binders display a Newtonian behaviour (constant J_{nr} value) with increasing stress level at all temperatures and indicates that these binders have lower stress sensitivity. However, as stress level increases up to 28.6, 12.8 and 6.4 kPa at temperatures of 40, 60 and 70^{o}C, respectively, the base binder sensitivity toward stress applied becomes noticeable. Moreover, some of the data is missing because the sample was failed earlier before being tested at the stress level due to the soft binder. While for the control binder, it starts to show shear thinning behaviour (the J_{nr} value increase with the stress applied) at stress levels of 28.6 and 12.8 kPa for temperatures of 60 and 70^{o}C, respectively. It can be seen that the base binder shows its higher stress sensitivity earlier than the control binder due to the higher penetration grade (D'Angelo 2009a).

At all tested temperatures, it can be observed that the addition of 7% RWP causes an increase in the stress sensitivity of the base binder. The RWP-modified binders reveal their higher stress sensitivity at stress levels as low as 0.1 kPa and become more significant as the test temperature increases. Among the three types of RWP used to modify base binder, the PP-modified binder exhibits the most sensitivity to the stress level as evidenced of a sudden increase of J_{nr} value from lowest at low stress level to similar with other RWP-modified binders at a higher stress level. The sudden increase in J_{nr} value becomes more significant when the temperature increased. D'Angelo (2009a) reported that at the low stress level with the binder still in its linear viscoelastic range, a stiff modified binder is formed due to the good intermolecular interaction between polymer and base binder. The polymer

network may not able to maintain the intermolecular interaction with base binder at higher stress conditions and thus, the polymer tends to show nonlinear response to high strain (Zoorob et al. 2012). Although the RWP-modified binders have higher stress sensitivities at all stress level, the value of J_{nr} of these binders were lower than the base binder and the control binder and thus, more rutting resistant is demonstrated by RWP-modified binder.

The SBS-modified binder exhibits minor increase in J_{nr} value and as the test temperature is raised and the stress level increased, the nonlinear behaviour of J_{nr} value is observed. Moreover, Figures 6.13 to 6.14 depicted that the RWP-modified binders have strong dependency of J_{nr} value to the stress applied compared to SBS-modified binder. Despite having lowest J_{nr} value at higher temperatures and stress levels, the rutting performance of SBS-modified binder may be similar to the RWP-modified binder if subjected to unexpected traffic loading at higher temperatures of road pavement because of it higher sensitivity upon stresses at 3.2 kPa and 70^{o}C.

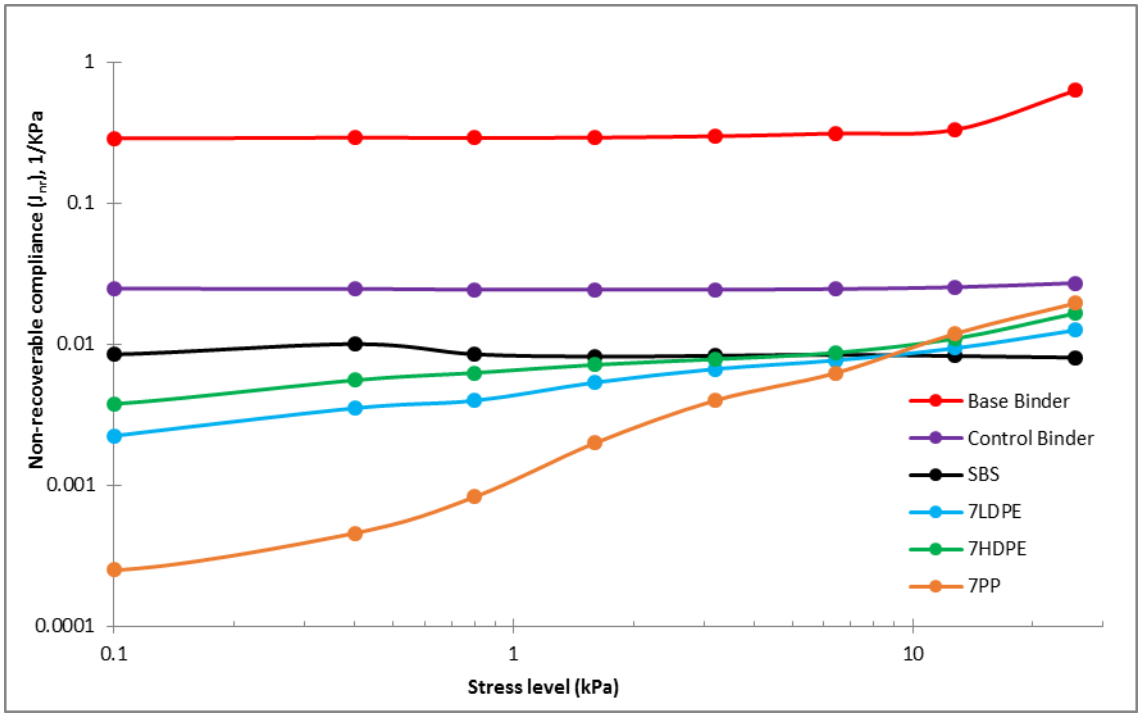


Figure 6.19: Stress sensitivity at 40°C

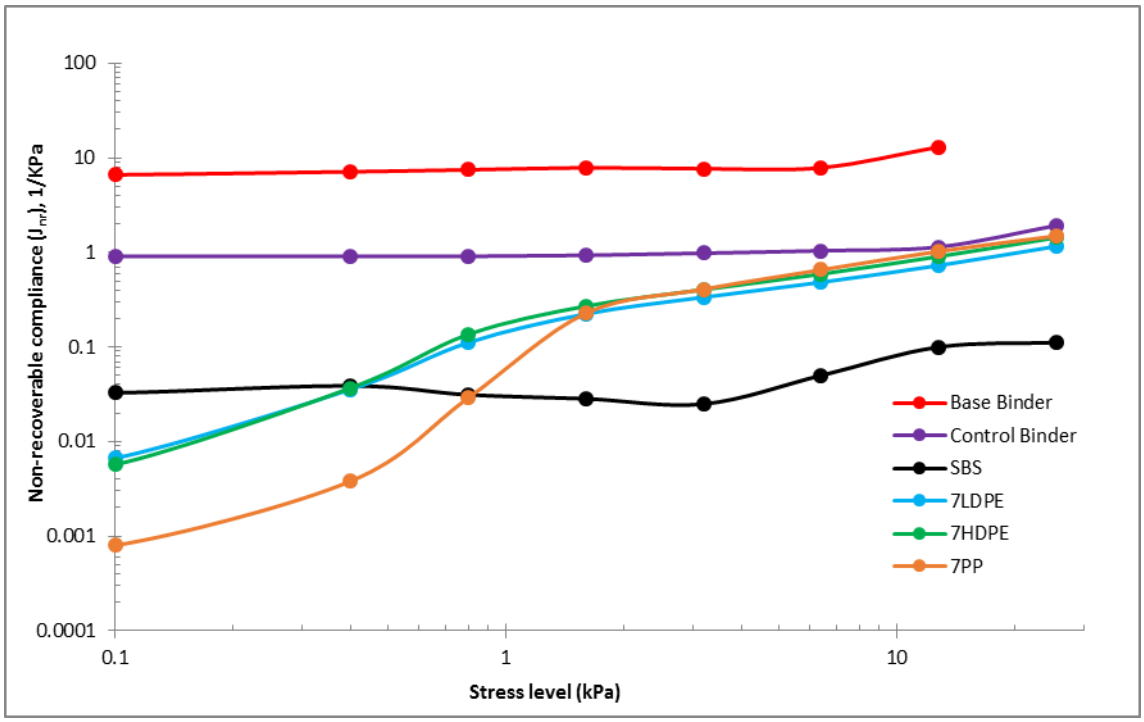


Figure 6.20: Stress sensitivity at 60°C

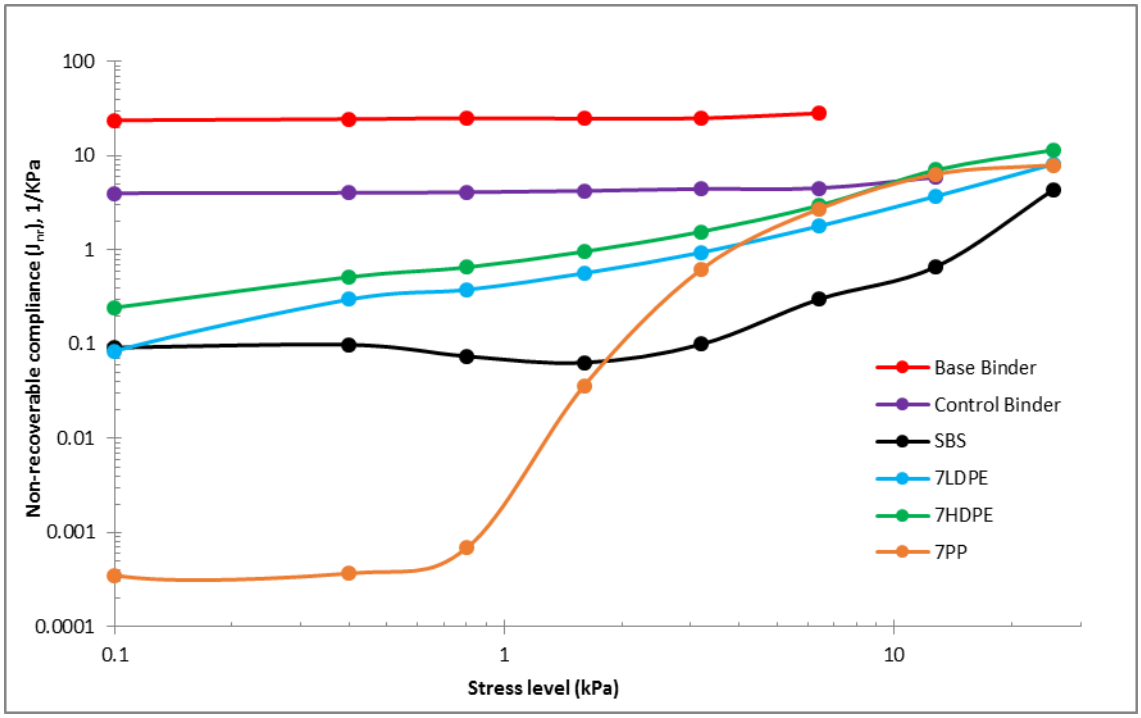


Figure 6.21: Stress sensitivity at 70°C

6.6 Summary

The 7% RWP-modified binders have been produced from the base binder with a standard of 160/220 penetration grade bitumen and then compared with its base binder, control binder consisting of a standard 40/60 penetration grade bitumen which represents the similar grade of penetration with 7% RWP-modified binders and lastly, a ready-made modified bitumen which is a SBS-modified binder. The mechanical properties and performance of these binders are compared and summarize as follow:

1. The results from the mechanical tests have shown that 7% RWP-modified binders may perform better in the field than the control binder although having similar grade of penetration due to the lower temperature sensitivity of the modified binder. In comparison with SBS-modified binder, both modified binders demonstrate similar improvement.

2. At high temperatures and low frequencies, in comparison with base and control binders, modified binders have significant effect on flow and deformation characteristics in which the binders exhibit elastic behaviour with better stiffness while base and control binder showed completely viscous behaviour with lower stiffness. Similar characteristics have been found for all studied binders at low temperatures and high frequencies with higher stiffness in term of complex modulus with a shift towards viscous behaviour.
3. The 7% RWP-modified binders showed better performance in rutting while the SBS-modified binder exhibited good fatigue cracking resistance.
4. From the LAS test:
    - In the case of polymer modified binder, the strain level from 1% to more than 30% should be tested in order to evaluate the damage evolution.
    - The RWP-modified binders had higher fatigue lives than other binders until levels of strain of 9% and the SBS-modified binder showed better fatigue lives at strain level more than 9%.
    - However, the RWP-modified binders have higher decreases in fatigue life than the SBS-modified bitumen with fatigue lines decreasing steeply for RWP-modified binders for all RWP types compared to SBS-modified bitumen at all temperatures.
    - At particular strain levels namely 2.5% and 5%, the RWP-modified binders showed better fatigue lives than other binders and thus, the effect in improving fatigue performance is pronounced in thick and thin asphalt pavements for these binders.

- Among the RWP-modified binders, the fatigue lives of 7LDPE-modified bitumen are higher than other binders at testing temperatures of 15 and 25^{o}C while 7HDPE exhibited the highest fatigue performance at temperature of 20^{o}C.
- The LAS ranking at test temperature of 20^{o}C and strain levels of 2.5 and 5% is as follow:

  7HDPE > 7LDPE > 7PP > SBS-Modified Binder > Control Binder > Base Binder

5. From the DENT test:
   - The polymer additive has significant influence on fracture properties of the binder.
   - Although 7% RWP-modified binders had lower value of specific work of fracture in comparison to the control binder at test temperature of 20^{o}C, the yielding capacity of 7% RWP-modified binders may be considered better than the control binder at low temperatures as evidenced by the specific work of fracture of control binder which could not be recorded as the sample is too brittle at temperature of 10^{o}C.
   - At both temperatures (10 and 20^{o}C), addition of 7% RWP resulted in significant increase in specific total work of fracture for base binder while the SBS-modified binder had highest specific total work of fracture among all tested binders.
   - Increase in temperatures resulted in rise and fall of the CTOD and w_e values, respectively. Temperature increase makes the binders less stiff and able to stretch more.

- Making comparison with the control binder and base binder, the 7% RWP-modified binders are susceptible to fracture at both testing temperatures, indicating that the addition of RWP resulted in poor fatigue resistance.
- A clear ranking can be observed among RWP-modified binder at both testing temperatures based on the CTOD value and at both testing temperatures, the 7HDPE-modified bitumen has higher strain tolerance followed by 7LDPE-modified bitumen with 7PP-modified bitumen showing the worst fracture resistance under the DENT test. However, as the temperature increases, the SBS-modified binder performed better in terms of fracture resistance than other binders.

6. Based on the MSCR result:
    - A general trend can be observed for all binders in which the higher the testing temperature and stress applied, the higher the J_{nr} values and the lower the percentage of recovery.
    - The results for two binders, namely base binder and 7HDPE-modified binder, displays a negative value for recovery particularly at higher levels of stress and temperature. The combination of high stress level with low modification level and tested at high temperatures are the cause of this situation.
    - Based on the desired parameter of rutting performance, namely J_{nr} values at a temperature of 40^{o}C, the overall ranking of the six binders is:

      7HDPE > 7PP > SBS > 7LDPE > Control Binder > Base Binder

    - The stress sensitivity of 7% RWP-modified binder and SBS-modified binder were higher in comparison with base binder and control binder as evidenced

by the shear thinning behaviour for J_{nr} values at low stress levels. However, the SBS-modified binder depicted stress sensitivities at test temperatures of 60 and 70^{o}C while the 7% RWP-modified binders show its sensitivity toward stress applied as early as 40^{o}C and become more significant as the temperature increases.

CHAPTER 7: **DURABILITY OF RWP-MODIFIED BINDER**

7.1 Introduction

This chapter deals with the study of durability of RWP-modified binder against moisture damage. The adhesion between binder and aggregate is vital to the performance of the asphalt mixtures in which the presence of moisture in asphalt mixture may degrade the cohesive resistance within the binder and the adhesive bond between binder and aggregates. In order to quantify the adhesion of binder-aggregate system, the Peel Test was used to evaluate the fracture properties regarding adhesive and cohesive bond by means of fracture energy.

For this purpose, all binders consist of the base binder, control binder, the RWP-modified binders at all modification level and SBS-modified binder were tested with the Peel Test in dry condition. Moreover, the effect of moisture damage against fracture strength is evaluated and only one modification level that yields higher fracture energy at dry condition is selected for testing with comparison of the base binder, control binder and SBS-modified binders. The test was performed at temperature of 20^{o}C and speed of 10mm/min. All results are analysed and compared in term of fracture energy and retained tensile force. Then, the type of failure is evaluated based on the fracture surface of the sample.

7.2 Fracture Energy in Dry Condition

Figure 7.1 depicted the average fracture energy for all binders at dry condition (unconditioned) and the range bars represent the maximum and minimum values for the replicates. Based on this figure, it can be seen that the base binder which is soft

has lower fracture energy than the control binder. The control binder is a hard binder with penetration grade of 40/60 in which more stiff than the base binder and thus, more energy is needed for breaking the cohesive bond apart (Zhang et al. 2016b).

Besides that, the addition of RWP into base binder increase the fracture energy compared to the base binder. These increases are more significant and higher for 7% RWP-modified binder while the 3 and 5% RWP-modified binder have almost the same fracture energy in the dry condition. The insignificant increase in fracture energy as a function of polymer concentration is caused by the conflict occurred between the surface tension ability to control the surface wetting and the binder strength to control the mechanical network in which the lower content of polymer in base binder tends to lose its wettability because of the polymer prefer to absorb the low molecular oil phase rather than increase the binder strength while the modification of base binder with higher polymer content may contribute to the improvement of the mechanical network that could increase the strength of the binder (Khattak et al. 2004). When the amount of RWP incorporate into base binder increases, the specific trend is observed among these three types of RWP namely at 5 and 7% of RWP used, the LDPE exhibited higher fracture energy with HDPE is second and lastly is PP. The sample prepared with 7% RWP have higher fracture energy compared to other modifications and thus, only 7% RWP-modified binders is considered and tested for moisture damage.

The comparison between similar penetrations binder showed that the fracture energy of control binder is higher than the RWP-modified binders due to higher stiffness value at test temperature of 20^{o}C as depicted in Figure 7.2. Only complex modulus of 7% RWP-modified binder is displayed in Figure 7.2 because the significant

increase of fracture energy of these binders in comparison with base binder could be observed for this modification compared to other modifications.

The highest average fracture energy is demonstrated by the SBS-modified binder although having lower stiffness value than the control and RWP-modified binders as shown in Figure 7.2 which indicate that the modifying binder with SBS polymer may improve the fracture resistance at dry conditions. This behaviour is equivalent with the relationship between stiffness modulus and tensile strength as depicted in Figure 7.3 in which the very high stiffness of bitumen at low temperature may exhibit lower tensile strength (Mo et al. 2011). Binder that is having lower tensile strength tend to yield lower fracture energy. Thus, even though the RWP-modified binders were stiff than the SBS-modified binder at tested temperature, it may be possible to had lower fracture energy than SBS-modified binder. The reason for higher fracture energy of SBS-modified binders has been reported by Zhang et al. (2017) in which the strongest resistance to self-cracking of the SBS-modified bitumen is due to the network structures of SBS polymer in which this polymer consists of styrene and butadiene block copolymers, the interlocking between these copolymers will form stable three-dimensional network structure that could enhance the cohesive properties of the binder. The lack of cross-linked structure in recycled RWP which belongs to plastomer group may contribute to lower tensile force and later yield lower fracture energy.

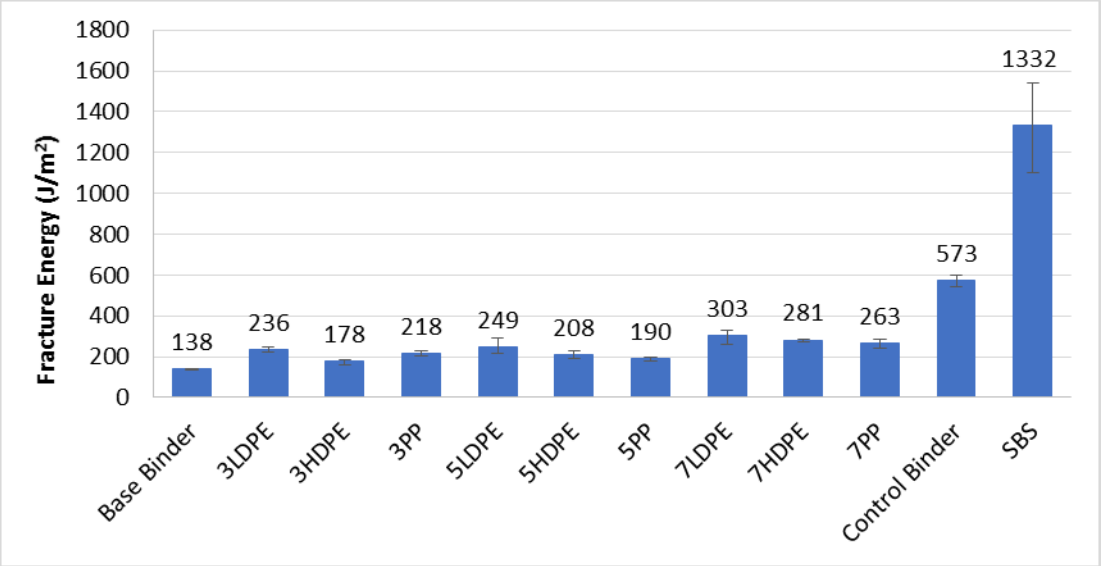


Figure 7.1: Fracture energy (J/m²) of aggregate-bitumen bonds for all binders in the dry condition

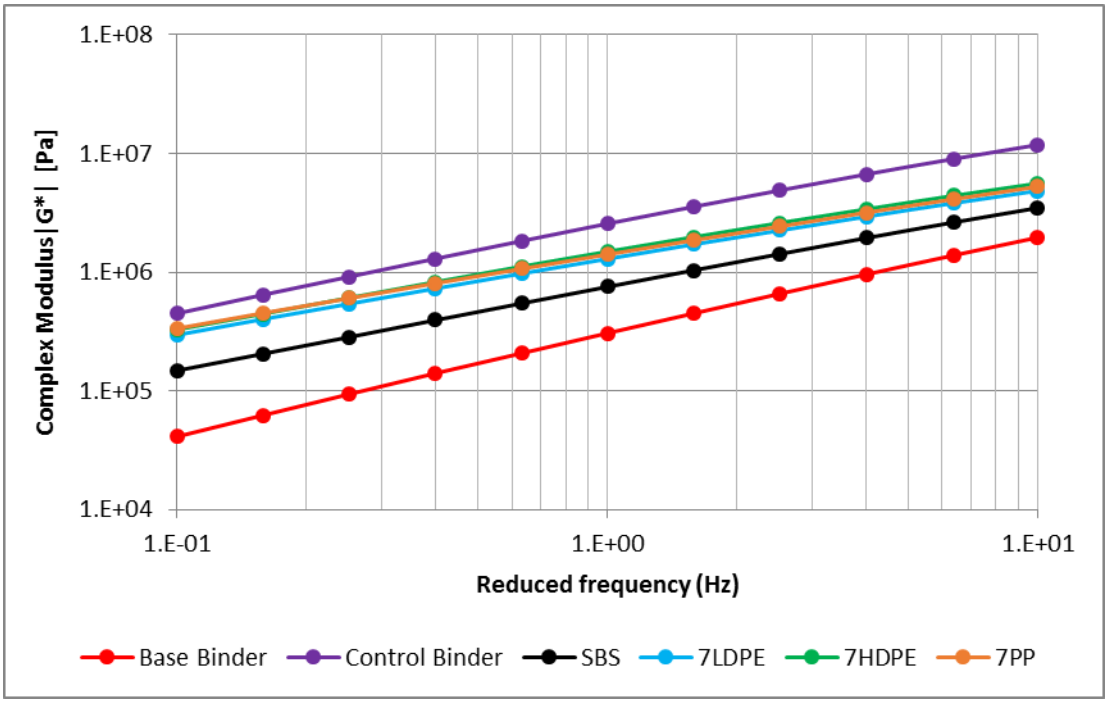


Figure 7.2: Complex Modulus of binders at temperature of 20°C

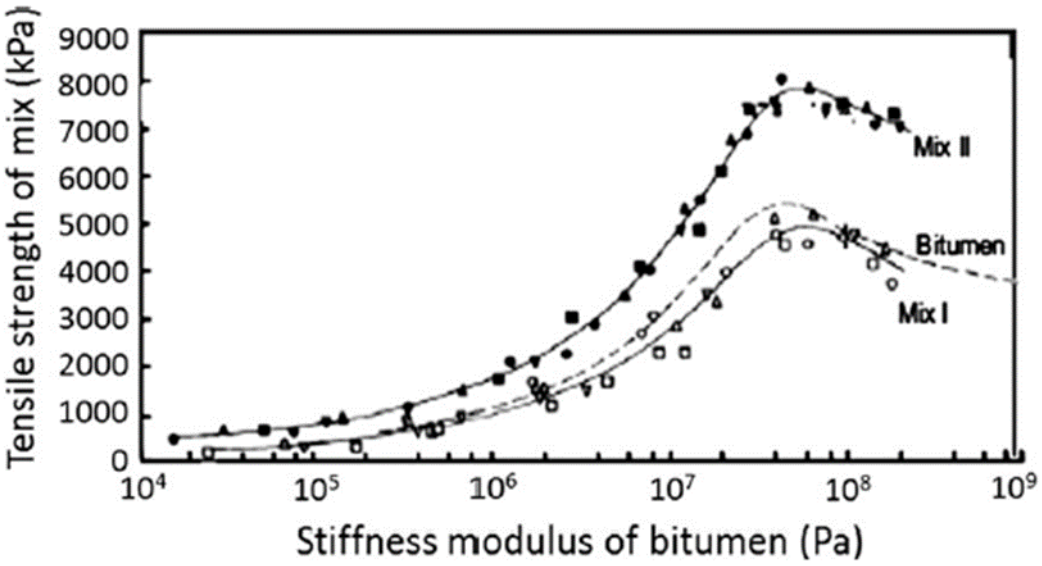


Figure 7.3: Bitumen stiffness modulus versus tensile strength reported by Heaukelom and Wijga (1973)

7.3 The Failure Mode at Dry Condition

Apart from fracture energy, the type of failure could be examined based on the fracture surface of the sample and the tensile load from the peel test (Zhang et al. 2016a) in which the higher the tensile load represents a cohesive (within the binder or same phase) failure while the adhesive failure tends to display a lower value of tensile load. However, in this study, the mode of failure is difficult to examine based on the tensile load particularly for RWP-modified binder because the complicated tensile load data is observed as demonstrated in Figure 7.4. The other RWP-modified binders depicted almost similar tensile force versus displacement curve. Thus, only the failure surface of sample prepared with twelve binders under dry condition are analysed for the evaluation.

Figure 7.5 displayed the failure surface achieved for all binders in dry condition. It can be seen that the bitumen layer is uniformly covered the surface of composite substrate for all tested binders. This indicates that the result of all binders under dry

condition exhibit similar failure surface mode namely a cohesive failure or in other words, the failure occurred is within the bitumen layer and thus, poor cohesion. This type of failure indicates that without the presence of moisture, the stronger bond is generated between the binder and aggregates rather than the binder itself.

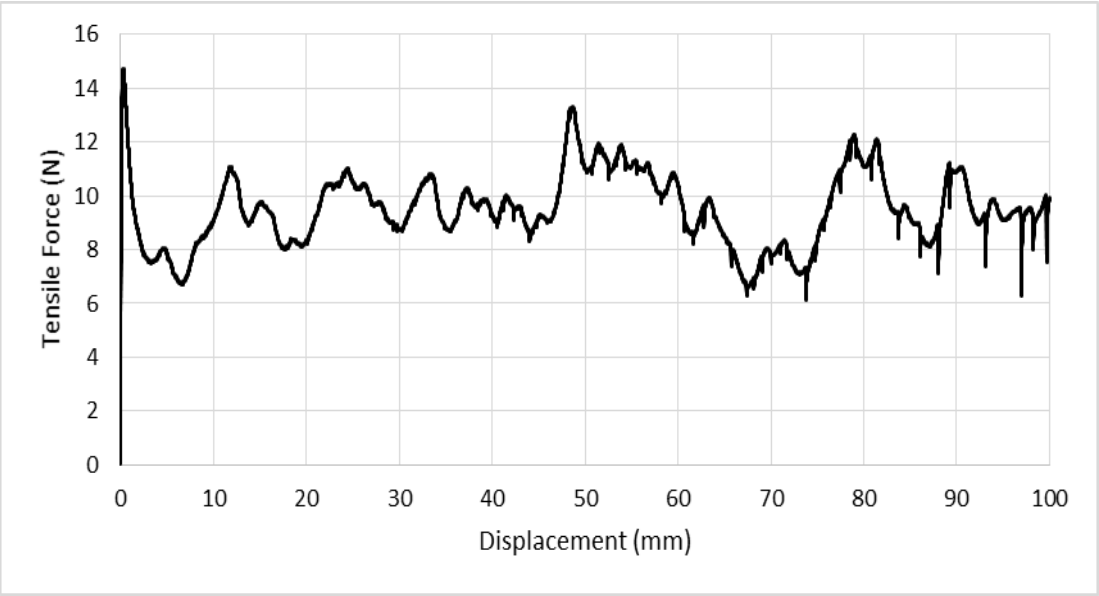


Figure 7.4: Tensile force versus displacement curve for sample prepared with 7LDPE-modified binder

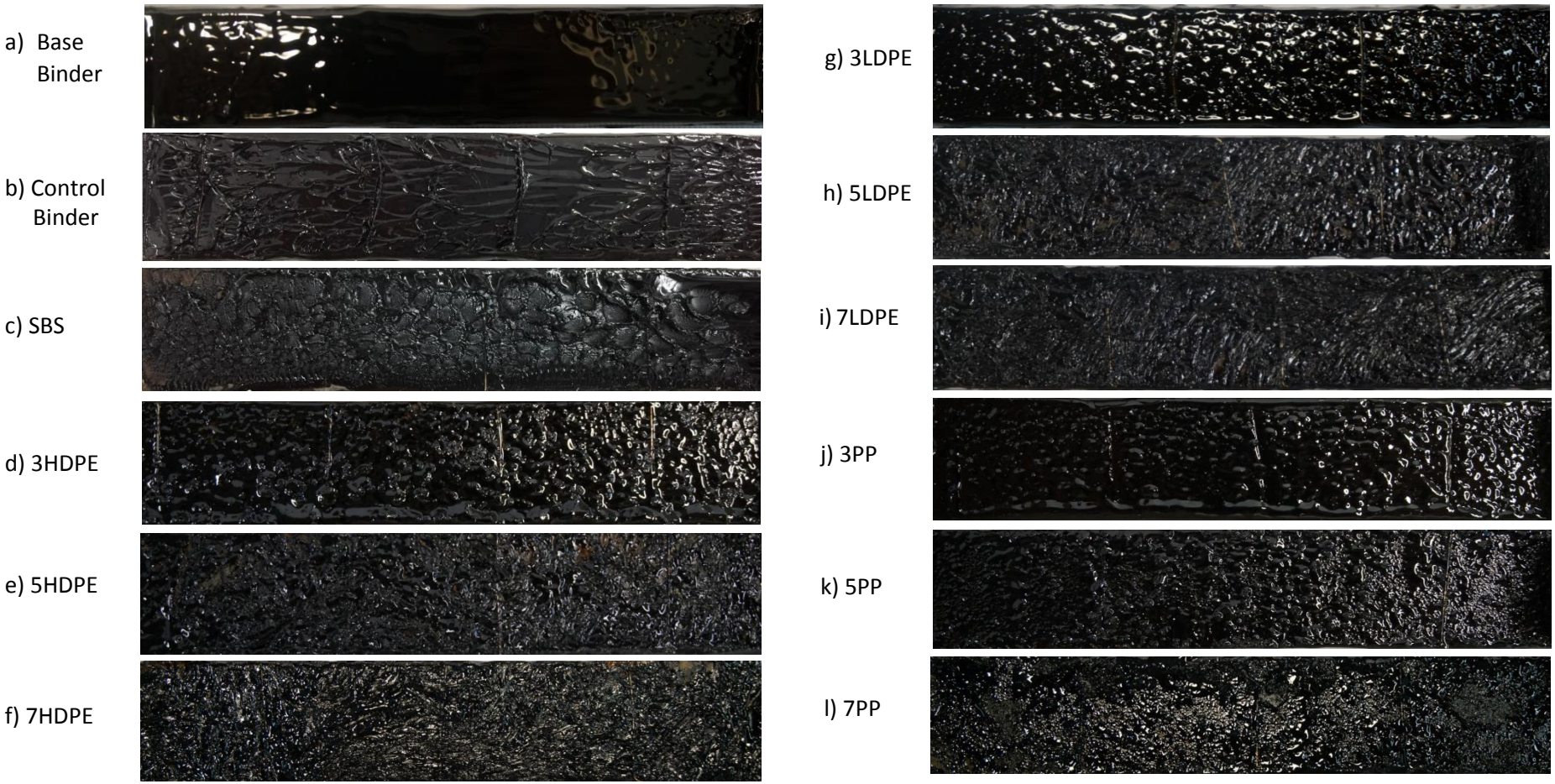


Figure 7.5: Failure surfaced of different binders under dry conditions at test temperature of 20^{o}C

7.4 The Effect of Bitumen After Moisture Conditioning

The whole samples were immersed in water for 14 days and 28 days at 20^{o}C before testing to investigate the effect of moisture on the bond between bitumen and aggregate system. The sensitivity of the different bitumen film with similar aggregate (granite) against moisture is evaluated by means of fracture energy for unconditioned and conditioned binders.

The results of the average fracture energy of different binders before and after moisture conditioning for 14 and 28 days at temperature of 20^{o}C is shown in Figure 7.6 and the minimum and maximum value of the replicates is represented by the range bar. It can be clearly seen that all binders experienced an increase in fracture energy after 14 days conditioning or in other word, the moisture durability of all binders are increase. This indicates that the presence of moisture may strengthen the bond system as a function of conditioning time. In term of cohesive bond, a study has been conducted by Figueroa et al. (2013) for investigating the effect of moisture damage on the virgin binder for a period of up to nine months and the results showed that the change in rheological properties of the binder is significant after conditioning time in which the increase in moisture conditioning causes the binder becomes stiffer and conclude that this phenomenon is similar to an oxidative ageing. While for adhesive bond, a good adhesion between the bitumen film and aggregate is produced if the bitumen has more carboxylic acids and sulfoxides (Petersen & Plancher 1998). The carboxylic acids and sulfoxides in bitumen can form strong chemical bonds with aggregate particularly granite due to the high contains of Si and Al (Zhang et al. 2016b). More component of carboxylic acids and sulfoxides produced after ageing due to volatilization and oxidation of the light component of bitumen

materials. Consequently, the more tensile force is needed to break the adhesive and cohesive bond apart and thus, increased the fracture energy.

Then, when the samples were conditioned in water up to 28 days, these binders exhibit different trend of fracture energy in which the base binder and 7LDPE-modified binder had a reduction in this value while the 7HDPE-modified binder, 7PP-modified binder, control binder and SBS-modified binder show some increment in the fracture energy. However, the changes of fracture energy are insignificant as evidence of the value of fracture energy after 28 days conditioning that shown by the range bar is overlapped with the range bar of fracture energy after 14 days submerged in water.

Based on the results after moisture condition, it appears that the effect of ageing on moisture sensitivity of bitumen and aggregate bond is maximal compared to the bitumen effects.

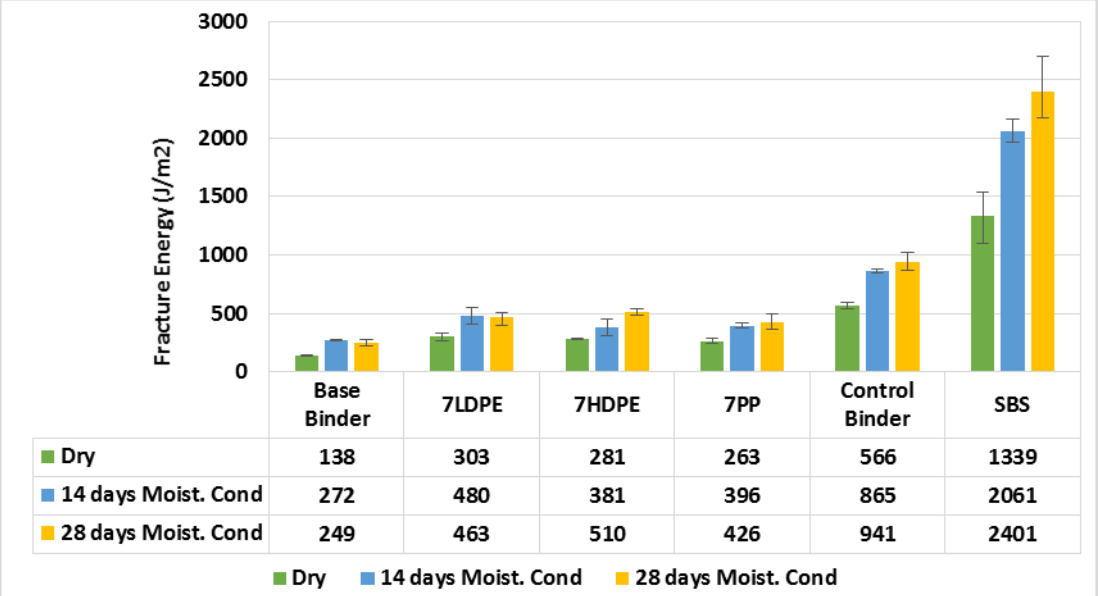


Figure 7.6: Fracture energy of binders under dry and moisture condition for 14 days and 28 days at test temperature of 20°C

a) Base Binder

Dry



14 days



28 days



b) Control Binder

Dry



14 days



28 days

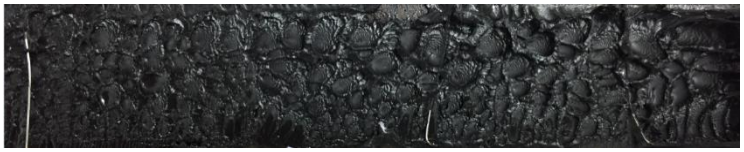


c) SBS-modified binder

Dry



14 days



28 days



Continue…

d) 7LDPE-modified binder

Dry



14 days



28 days



e) 7HDPE-modified binder

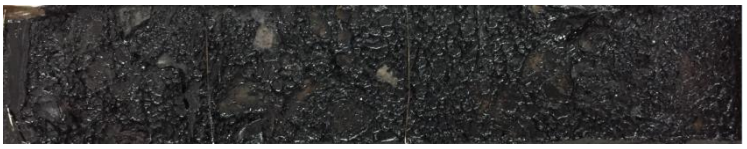
Dry



14 days



28 days

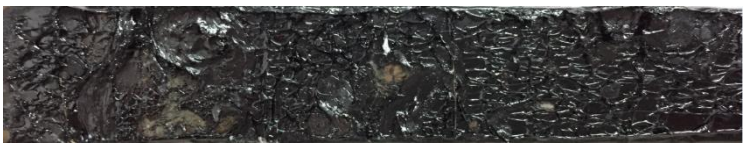


f) 7PP-modified binder

Dry



14 days



28 days



Figure 7.7: Failure surfaces of sample in dry and after 14 and 28 days moisture condition prepared with, a) Base Binder, b) Control Binder, c) SBS-modified Binder, d) 7HDPE, e) 7HDPE and f) 7PP.

The type of failure could be analysed based on the observation of the failure surface of the samples. Figure 7.7 is depicted the fracture surfaces of samples prepared with base binder, control binder, SBS-modified binder and all 7% RWP-modified binders before and after moisture condition of 14 and 28 days. As been mentioned in previous section, cohesive failure occurred for all binders at dry condition. After being immersed for 14 and 28 days in water, the results of base binder and 7LDPE-modified binder are obviously displayed 100% of cohesive failure in which this failure occurs within the bitumen layer and after testing, the bitumen layer is still covering the aggregate surface. This means that the adhesion bond between bitumen and aggregate is stronger than the bond in the bitumen itself after moisture conditioning. The other binders showed different failure surfaces. Under moisture condition of 14 days, a cohesive failure occurred for the SBS-modified binder and when the moisture conditioning time increase up to 28 days, this binder tends to change from cohesive failure to adhesive-cohesive mix failure with the adhesive failure appeared relatively small compared to the cohesive failure. The control binder, 7HDPE and 7PP-modified binder show an adhesive-cohesive mixed failure after 14- and 28-days moisture condition with the dominant mode of failure surfaces was cohesive. Table 7.1 summarised the failure mode for all samples of binder for dry and wet condition.

The retained fracture energy of all binder at all conditions showed in Table 7.2. The retained fracture energy represents the moisture sensitivity of the sample in which could be used to quantify the loss of the sample strength due to the moisture damage in the bitumen and aggregate system or the moisture sensitivity (Blackman et al. 2013; Cui et al. 2014). Generally, the larger magnitude of retained fracture energy of the sample would lead to the better resistance to fracture.

It can be seen that the retained fracture energy increased with the increase in conditioning time for sample prepared with the control binder, SBS-modified bitumen, 7HDPE and 7PP-modified binders after being submerge in water for 14 and 28 days, showing the failure properties of bitumen and aggregate joint is not depending on the effect of moisture but could be attributed to ageing in which the binders become more stiff as a function of water conditioning time and hence, more force is needed to break the bond. Figueroa et al. (2013) immersed a thin films of binder in water up to 9 months and find out that the increase in bond strength due to the increase in the binder stiffness is contributed by the water during the moisture conditioning time in which the similar effect is observed for oxidative ageing of the bitumen. A study has been performed earlier by Airey et al. (2005) on the asphalt mixture regarding the effect of combined ageing and water sensitivity on the stiffness, it shows the initial stiffening effect is depicted by the asphalt mixture after undergoing the moisture conditioning and ageing regimes but then further various moisture sensitivity and ageing regimes make the stiffness decreased.

The other binders namely base binder and 7LDPE-modified bitumen demonstrated a similar trend namely the retained fracture energy increase up to 96% and 59% of that of the dry sample after 14 days moisture conditioning, respectively. However, further conditioning time up to 28 days showed that the retained fracture energy is reduced about 16% and 6% for base binder and 7LDPE-modified bitumen, respectively from the previous moisture conditioning.

By referring to Tables 7.1 and 7.2, after 14 days of moisture condition, the retained fracture energy of the sample is increased while the failure surface of the all samples are different among each other. Nevertheless, as the conditioning time increase to 28

days, an increment of retained fracture energy is exhibited for a sample that is having a mix failure mode (adhesive and cohesive failure) with cohesive failure is dominating the fracture surface as evidence of more bitumen residue remaining on the aggregate and only a fraction of the aggregate surface could be seen. While the other two binders which had a reduction in retained fracture energy are the base binder and 7LDPE-modified bitumen demonstrated the cohesive failure surface.

The relationship between tensile load and surface failure has been done by Zhang et al. (2016a) in which reported that a higher tensile load is represented by the cohesive failure while the lower of tensile load is displayed by the adhesive failure and the tensile load is correlate well with the fracture energy and thus, the adhesive failure tends to exhibit lower fracture energy and the cohesive failure results in a higher energy. However, by referring Table 7.1, the result found from this study demonstrates the opposite as evidence of the tensile load of all binders displayed in Table 7.3 showed some increment as a function of moisture conditioning time although the failure mode is adhesive-cohesive mixed failure while after 28 days of moisture conditioning time, the cohesive failure mode exhibits a slightly decrease particularly for base binder and 7LDPE-modified bitumen. Hence, it is speculated that the increases in tensile force in the Peel test which yield increase in the fracture energy of samples after being exposed to moisture for a certain period of time is may cause by the stiffening effect of bitumen due to the ageing during moisture conditioning in which this effect is more dominant than the effect of moisture condition and binder type.

Table 7.1: Failure mode of samples from direct observation

Binder	Type of Failure		
	Dry	14 days Moisture Condition	28 days Moisture Condition
Base Binder	Cohesive	Cohesive	Cohesive
Control Binder	Cohesive	Mix (Mainly cohesive)	Mix (Mainly cohesive)
SBS	Cohesive	Cohesive	Mix (Mainly cohesive)
7LDPE	Cohesive	Cohesive	Cohesive
7HDPE	Cohesive	Mix (Mainly cohesive)	Mix (Mainly cohesive)
7PP	Cohesive	Mix (Mainly cohesive)	Mix (Mainly cohesive)

Table 7.2: Retained fracture energy of all binders

Binder	Retained Fracture Energy (%)		
	Dry	14 days Moisture Condition	28 days Moisture Condition
Base Binder	100	196	180
Control Binder	100	153	166
SBS	100	154	179
7LDPE	100	159	153
7HDPE	100	136	181
7PP	100	150	162

Table 7.3: Tensile force of all binders

Binder	Average Tensile Force (N)		
	Dry	14 days Moisture Condition	28 days Moisture Condition
Base Binder	4.8	8.0	7.5
Control Binder	17.1	24.2	25.9
SBS	33.3	48.7	55.8
7LDPE	10.1	14.5	14.1
7HDPE	9.5	12.2	15.4
7PP	9.2	12.6	13.3

7.5 Summary

1. At dry condition, all binders exhibited cohesive failure with the addition of 7% RWP into base binder show better resistant to fracture compared to base binder while the SBS-modified binder and control binder had higher fracture energy than the 7% RWP-modified binder. Then, RWP-modified binders with higher polymer content were selected for the further moisture conditioning test together with the base, control and SBS-modified binder.
2. Improvement in moisture durability for all binders after conditioning show that the effect of stiffening is dominated in which ageing take place during the conditioning based on previous study and the degree of improvement is vary depending on the types of binder.
3. The increase in fracture energy is demonstrated by the cohesive failure due to higher tensile force. The opposite relationship was observed compared to the previous study done by Zhang et al. (2016a) between the tensile force and the fracture surface of the sample namely in this study, further conditioning shows an

increment in fracture energy although adhesive failure has occurred on the sample surface.

4. The magnitude of fracture energy in dry and wet condition was found to be bitumen dependent in which the SBS-modified binder has higher fracture energy for all conditions although the complex modulus of this binder is lower than the control binder and all 7% RWP-modified binders which may due to the strongest resistance to self-cracking of the SBS polymer.
5. The fracture energy of the binders in all conditions is influenced by the nature of network of the binder itself.

CHAPTER 8: PERFORMANCE OF RWP-MODIFIED BINDER IN ASPHALT MIXTURE

8.1 Introduction

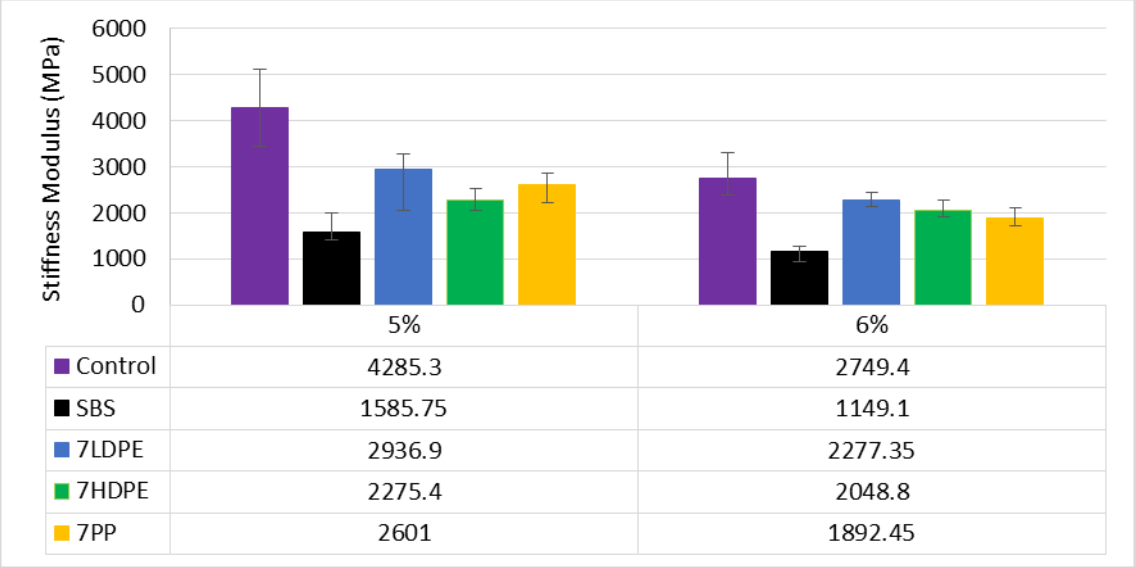
Based on the rheological and performance tests of binders, the base binder with the highest content of all RWPs namely 7% showed most promising result and thus, these binders were selected for the production of asphalt mixture. Besides that, two conventional binders were used for comparison which are the control binder (penetration grade 40/70) that is lies in the similar penetration grade of RWP-modified binder and the SBS-modified binder as the ready-made binder from the supplier. Those binders were used for manufacturing asphalt mixture and tested for performance properties of fatigue cracking and rutting that is normally occurred at intermediate (10-40^{o}C) and high temperatures (more than 40^{o}), respectively. The performance of asphalt mixtures were characterised in term of the stiffness modulus, resistance to fatigue and resistance to rutting by means of ITSM, ITFT and RLAT, respectively with two binder contents namely 5% and 6% for comparison.

8.2 Indirect Tensile Stiffness Modulus (ITSM) test

The result of ITSM at testing temperature of 20^{o}C were obtained from the average of ten samples for each mixture shown in Figure 8.1 and the range bar represent the maximum and minimum value of sample replicates. The average stiffness modulus values are depicted as a function of the binder content in asphalt mixture.

It can be seen that as the binder content increased, the stiffness modulus value for each mixture is decreased significantly. Among the tested binders, a clear trend of stiffness modulus value could be observed with the control binder has relatively higher

stiffness modulus for both bitumen content in the mixture followed by the RWP-modified mixture and lastly, SBS-modified asphalt mixture. As regards the asphalt mixtures produced with RWP-modified binders, it can be noticed that the 7LDPE-modified mixture depicted greater stiffness compared to 7HDPE and 7PP for both bitumen content. The higher stiffness modulus value of asphalt mixture indicates the ability of this mixture to spread the load is higher and will become more resistance to asphalt pavement distresses (Bala et al. 2018). Lower stiffness asphalt mixture tends to have better resistance to fatigue (Maupin 1971; Modarres & Hamedi 2014). However, in term of rutting, the stiffer asphalt mixture will experience more rutting when subjected to dynamic loading while conversely occurred under the static loading (Moghaddam et al. 2014). Thus, the performance test of asphalt mixture is performed and the results were discussed in the next sections.



	5%	6%
Control	4285.3	2749.4
SBS	1585.75	1149.1
7LDPE	2936.9	2277.35
7HDPE	2275.4	2048.8
7PP	2601	1892.45

Figure 8.1: ITSM result for the different asphalt mixtures at temperature test of 20°C

8.3 ITFT Test Result

The measurement of fatigue life of each mixtures is carried out by means of ITFT test and the prediction equation of fatigue life was obtained by using the regression analysis.

The result of fatigue tests that performed at 20^{o}C for asphalt mixture with binder content of 5 and 6% were shown in Figures 8.2 and 8.3, respectively. The first noticeable result of fatigue test is the highest fatigue lives of SBS-modified mixture in comparison to the control and RWP-modified mixture at both content of the binder. In fact, the good fatigue resistance of this mixture caused of the presence of SBS polymer in which has been proven by many previous studies (Chen et al. 2002; Airey 2004; Kumar et al. 2006; Saboo & Kumar 2016; Vamegh et al. 2019).

Moreover, better fatigue life is demonstrated by the RWP-modified mixtures compared to the control mixture at strain level particularly above 300 and 450 με for 5 and 6% binder content, respectively. Among the RWP-modified mixtures, the fatigue resistance of 7LDPE-modified mixture appears to be poor than 7HDPE and 7PP-modified mixture. The fatigue life of 7HDPE and 7PP mixture depicted almost similar and difficult to interpret as the lines are overlapping to each other which indicates that the behaviour of these two RWP upon fatigue is similar. Thus, the prediction of fatigue life at strain of 100 με and strain at 10^6 cycles obtained from the fatigue function is used to make the comparison and shown in Table 8.1. The R^2 values developed from fatigue equation for all mixtures are above 0.97, meaning that a high degree of accuracy is depicted by the fatigue function relationship between fatigue life and initial strain.

In term of the effect of binder content, it can be seen that the result found in this study is equivalent with the previous study done by Nejad et al. (2010) in which the fatigue life of mixtures containing 6% of binder shows better fatigue performance compared to binder content of 5% as evidence of higher fatigue life at strain level of 100 με for all mixtures. Increase in fatigue live is depicted by the RWP-modified mixtures although the increment is insignificant when the binder content in asphalt mixture

increased, meaning that the effect of binder content with the addition of RWP-modified binder in asphalt mixture is insignificant. Besides that, no substantial improvement of fatigue lives is recorded for the RWP-modified mixtures, indicates these mixtures might not able to perform well on fatigue.

The slope of the curve from fatigue equation could be used to evaluate the mixture susceptibility against the change of strain level (Saboo & Kumar 2016) in which highest susceptibility to the change of strain level is indicated by the higher absolute slope value. According to Table 8.1, it can be seen that the SBS-modified mixtures displayed highest susceptibility to the strain level, followed by control mixture while the RWP-modified mixture showed least susceptible as evidence of lower absolute slope value and as the binder content increased, the trend is more noticeable.

For both binder content, a similar ranking of fatigue resistance at temperature of 20^{o}C is obtained as follow:

SBS > Control > 7PP > 7HDPE > 7LDPE

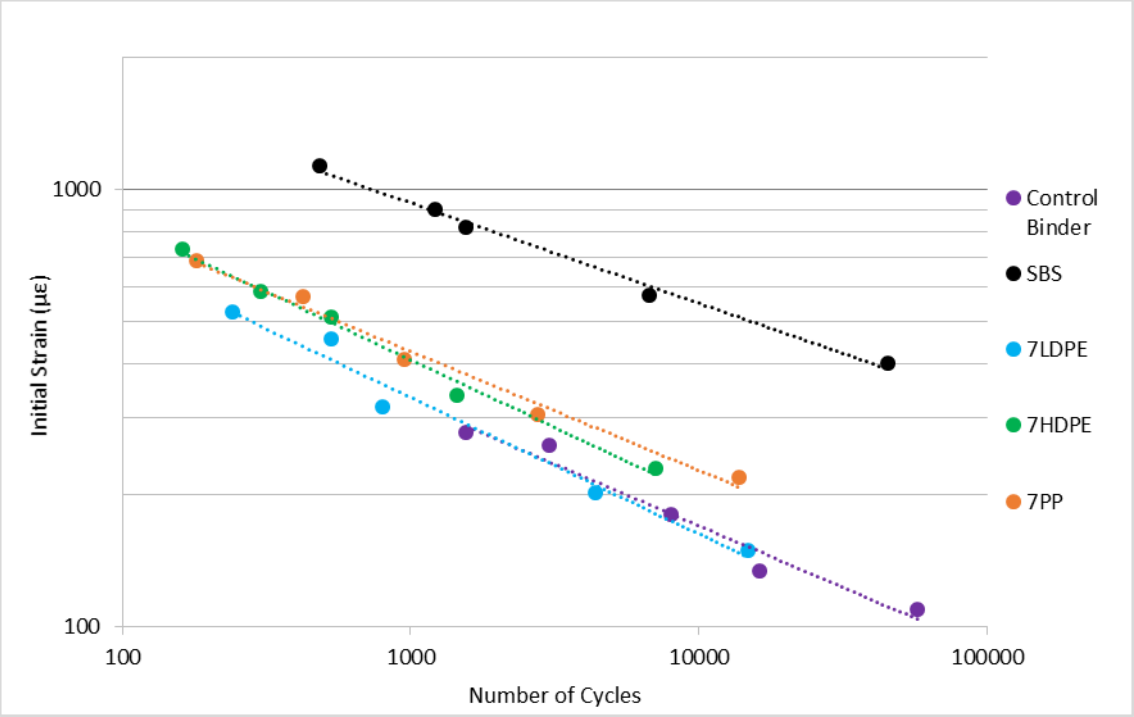


Figure 8.2: Fatigue line of studied mixes for 5% binder content

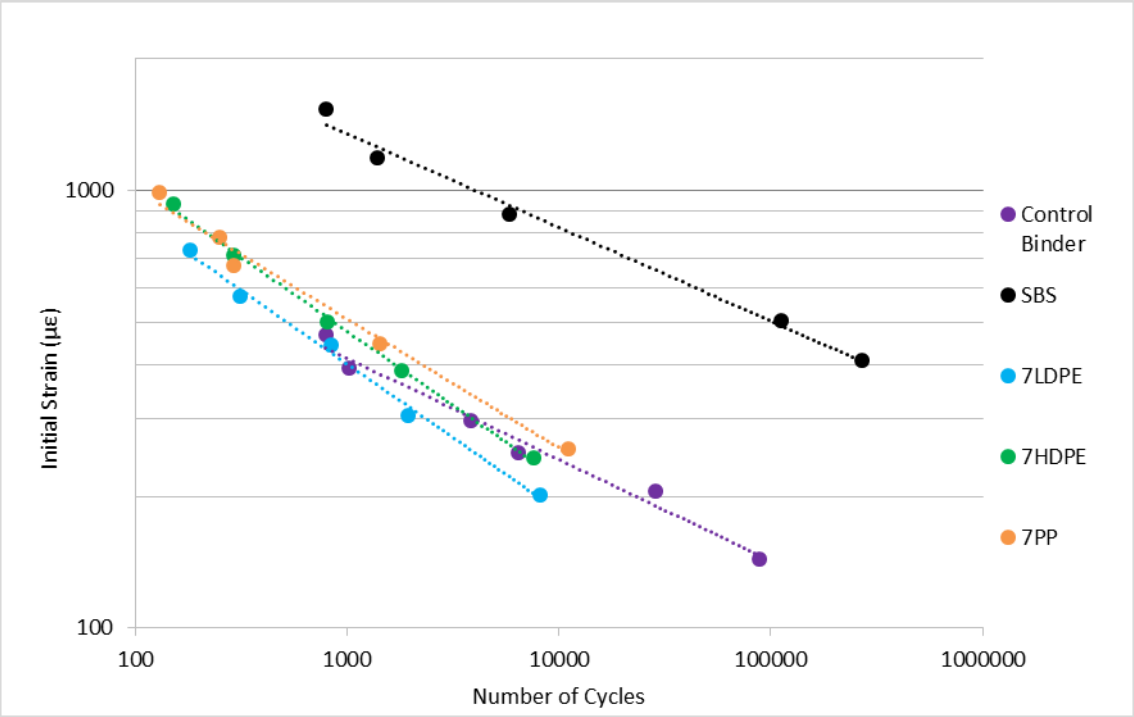


Figure 8.3: Fatigue lines of studied mixes for 6% binder content

Table 8.1: Fatigue parameters and relationships for asphalt mixtures

Binder Content	Binder	Strain @10^6 cycles (microstrain)	Fatigue cycles @100 microstrain	Fatigue Equation	Regression Coefficient (R^2)
5%	Control Binder	46	5.55E+04	$N_f = 4 \times 10^{11} \varepsilon^{-3.43}$	0.97
	SBS	192	1.57E+07	$N_f = 7 \times 10^{15} \varepsilon^{-4.33}$	0.99
	7LDPE	39	4.07E+04	$N_f = 7 \times 10^{10} \varepsilon^{-3.12}$	0.97
	7HDPE	48	7.82E+04	$N_f = 2 \times 10^{11} \varepsilon^{-3.20}$	0.99
	7PP	65	1.69E+05	$N_f = 3 \times 10^{12} \varepsilon^{-3.63}$	0.99
6%	Control Binder	84	3.30E+05	$N_f = 1 \times 10^{14} \varepsilon^{-4.24}$	0.98
	SBS	307	1.70E+08	$N_f = 3 \times 10^{17} \varepsilon^{-4.62}$	0.99
	7LDPE	39	6.29E+04	$N_f = 5 \times 10^{10} \varepsilon^{-2.95}$	0.99
	7HDPE	46	1.01E+05	$N_f = 8 \times 10^{10} \varepsilon^{-2.95}$	1.00
	7PP	66	1.95E+05	$N_f = 1 \times 10^{12} \varepsilon^{-3.36}$	0.99

8.4 RLAT Test Result

Figure 8.4 displays the results of RLAT test for control, SBS-modified and RWP-modified mixtures with binder content of 5 and 6% at tested temperature of 40°C. The parameter obtained from the RLAT test for evaluating the rutting performance of asphalt mixture is the creep curve that contain of the accumulative axial strain as function of the number of load cycles in which the lower potential to rutting resistance of the asphalt mixture is presented by the higher accumulative axial strain (Ali et al. 2017). Besides that, the result of axial strain from RLAT test is considered to be more susceptible to rutting when the axial strain is more than 2% (Widyatmoko 2008).

According to Figure 8.4, it can be seen that the cumulative axial strain of SBS and 7LDPE-modified mixture are below this threshold for both binder content. The other

mixtures which are control and 7PP-modified mixture are having poor resistance to rutting compared to other mixtures for both binder content as evidence of its axial deformation is exceeded 2%. Meanwhile, the 7HDPE-modified mixture tends to have an axial strain of more than 2% when the binder content is 5% while conversely happen for binder content of 6%.

Figure 8.4 clearly show that mixtures with 6% binder content had lower accumulated axial strain and therefore, more resistance to rutting except for control and 7PP-modified mixtures. However, previous studies reported that the susceptibility to rutting is generally more pronounced for mixtures containing higher binder content (Garba 2002; Tayfur et al. 2007; Gardete et al. 2008; Al-Khateeb & Basheer 2009) due to thicker binder film between aggregate particles may decrease the internal friction among the aggregates in which the binder will carry the loads more than the aggregate structures (Tayfur et al. 2007). On the contrary, higher binder content tends to exhibit less susceptibility to rutting in this study could be attributed to the improvement of recovery percentage of these binders from the MSCR test as shown in Figure 8.5 in which the recovery improvement is significant for SBS and RWP-modified binder due to the relative increase in polymer content of the mixture. Moreover, the control and 7PP-modified binder display almost similar recovery percentage (Figure 8.5) and thus, these two binders are having similar behaviour of rutting performance in the mixture in which the general effect of binder content in mixture as higher binder content may decrease rutting resistance are applicable for these mixtures. Furthermore, the addition of 0.5% soft binder above optimum binder content to mixture would make the rutting

susceptibility increase while an extra of 0.5% hard binder tends to improve the durability without affecting the rutting resistance (Brown et al. 2004).

Generally during the test, the mixture will undergo three stages of rutting deformation as shown in Figure 8.6. However, the result from the RLAT test in Figure 8.4 particularly for SBS and RWP-modified mixtures are not reached stage 3 and the discrepancy of total axial strain after 5400 pulses for some mixtures is difficult to distinguish due to the overlapping curve at the end of the pulses. This indicates that the polymer-modified mixtures could take a higher number of load repetitions without causing any damage. Thus, more loading pulses are required during the test for these mixtures exhibit the tertiary zone for the damage occurrence.

The minimum strain rate of the mixtures is depicted in Figure 8.7 in which this parameter represents the rate of change in the rutting performance of the mixtures (Azari & Mohseni 2013). As seen, mixture with SBS-modified binder has lowest minimum strain rate which means that the superior rutting resistance is displayed by this mixture as the change in rutting performance is almost negligible, followed by the RWP-modified mixtures and the control mixture demonstrated worst resistance to rutting as evidence of higher minimum strain rate, thus performance of rutting changes faster. Even though the RWP-modified mixtures appear to have higher minimum strain rate than the SBS-modified mixture, the better performance in rutting is demonstrated by these mixture compared to the control mixture that is having similar penetration grade of binder. As been mention earlier that mixtures with 6% binder content perform better in rutting and thus, only mixture containing this binder content is considered for the ranking of rutting resistance namely:

SBS > 7HDPE > 7LDPE > Control > 7PP

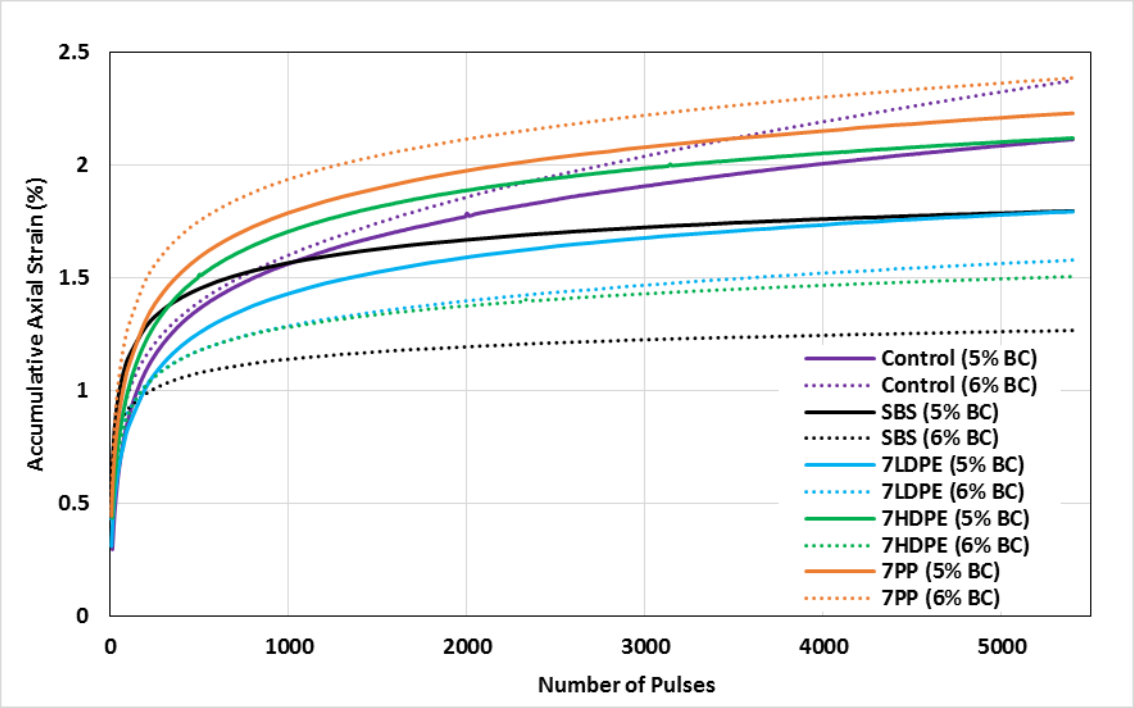


Figure 8.4: RLAT results of different mixtures tested at temperature of 40°C

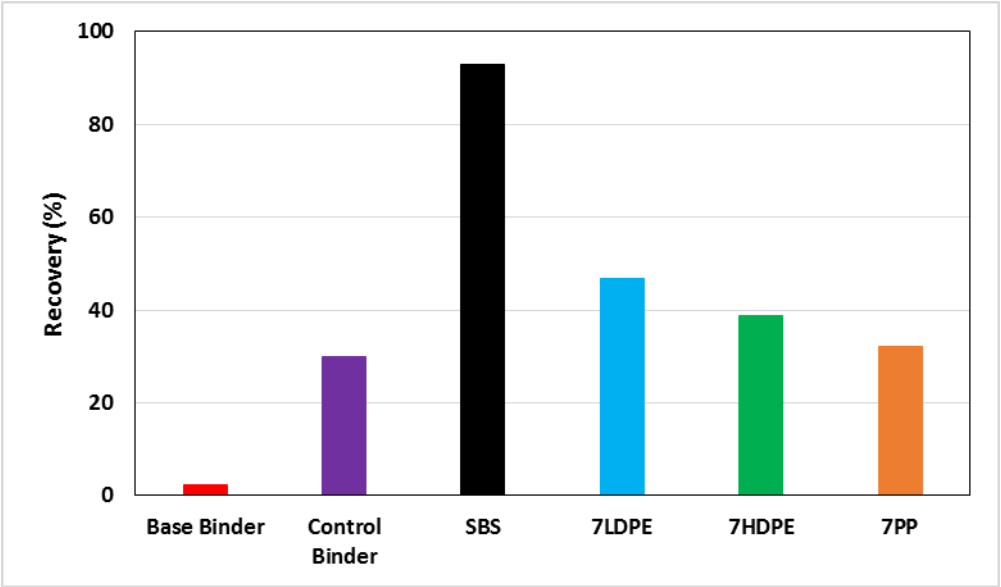


Figure 8.5: Recovery percentage of binders at stress level of 25.6 kPa and at temperature of 40°C

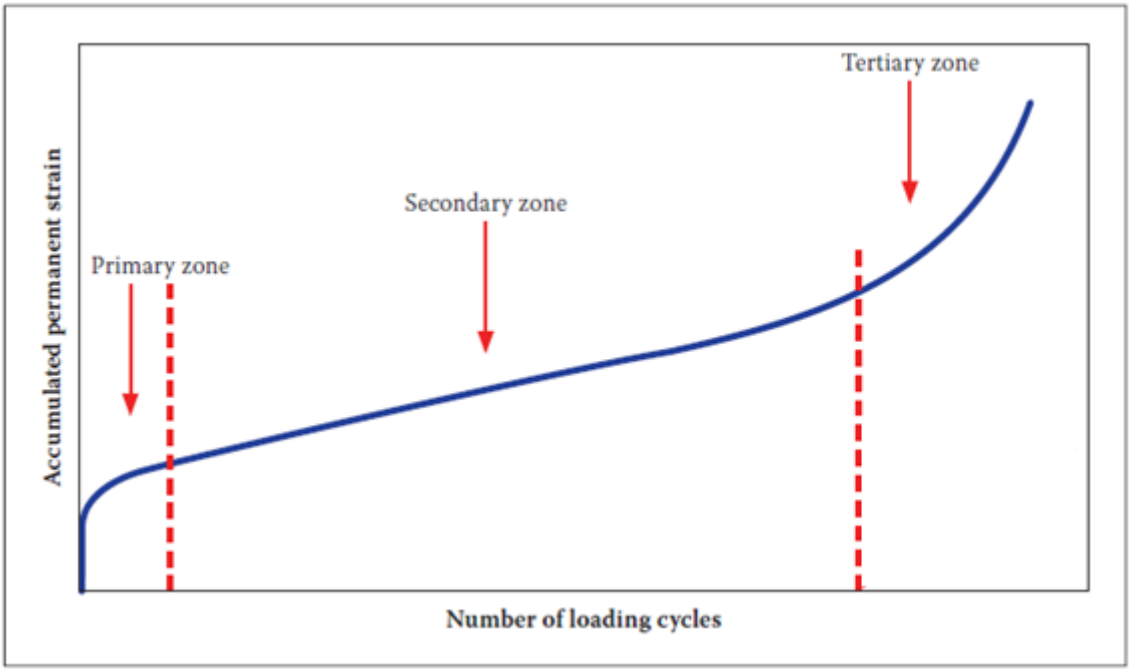


Figure 8.6: Typical three-stage curve of creep test result

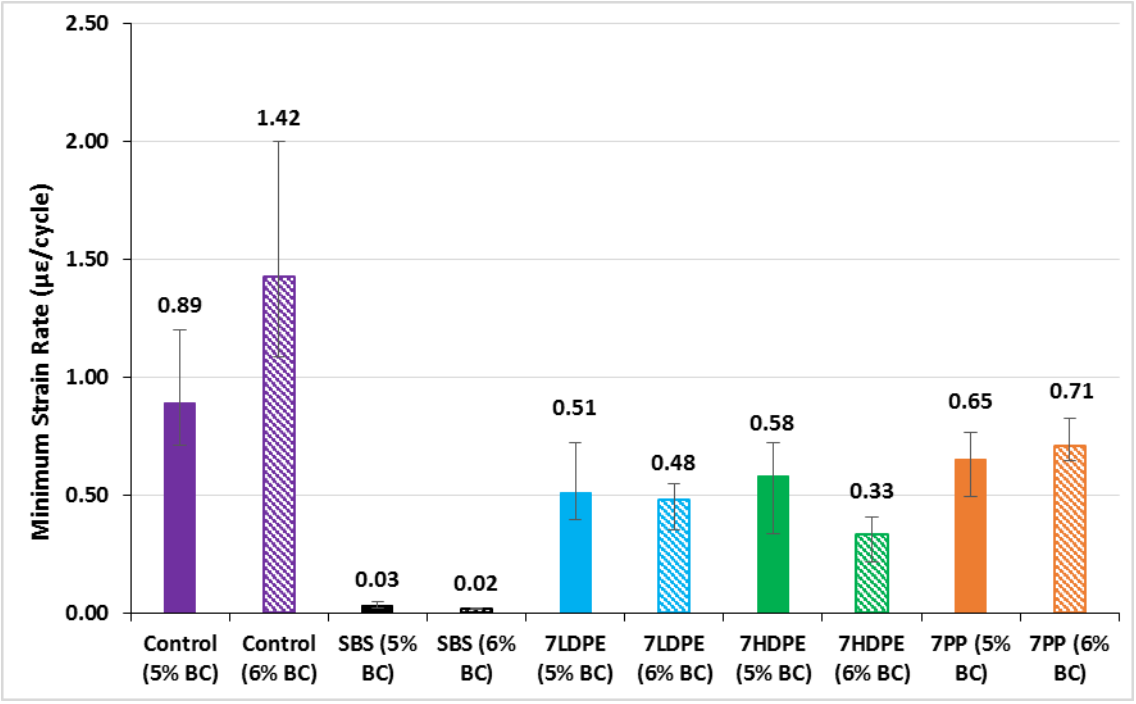


Figure 8.7: Result of minimum strain rate

8.5 Summary

1. ITSM:

   - For all mixtures, the stiffness modulus values are significantly decrease as the binder content increased.
   - The overall ranking of the different mixtures for both binder contents are similar namely:

     Control > RWP-modified binders > SBS

   - As regards the asphalt mixtures produced with RWP-modified binders, the 7LDPE-modified mixture had highest stiffness compared to 7HDPE and 7PP for both bitumen content.

2. ITFT:

   - Mixtures containing RWP-modified binders appear to be lower resistance to fatigue cracking than the SBS-modified binder and control binder.
   - The improvement in fatigue resistance is more pronounced as the binder content increased.
   - At strain level particularly above 300 and 450 με for 5 and 6% binder content, respectively, better fatigue life is demonstrated by the RWP-modified mixtures in comparison to the control mixture.
   - According to the slope of the fatigue life curve, the SBS-modified mixtures displayed highest susceptibility to the strain level, followed by control mixture while the RWP-modified mixture showed least susceptible.
   - Both binder contents exhibited similar ranking of fatigue resistance at temperature of 20^{o}C namely:

     SBS > Control > 7PP > 7HDPE > 7LDPE

3. RLAT:

- The plot of accumulation axial strain as a function of number of pulses from the RLAT test for SBS and RWP-modified mixtures are not reached stage 3, means that these mixtures could take a higher number of load repetitions without causing any damage and thus, better rutting resistance.
- The SBS-modified mixture exhibited slowest changing in rutting performance as evidence of lower value of the minimum strain rate.
- The mixtures with 6% binder content had lower accumulated axial strain and therefore, more resistance to rutting. Thus, the ranking of rutting resistance namely:

  SBS > 7HDPE > 7LDPE > Control > 7PP

Overall, based on the mixture tests conducted, the performance of the mixtures are significantly affected by the type of the binder used and binder content in the mixture.

CHAPTER 9: CONCLUSIONS AND RECOMMENDATIONS

9.1 Conclusions

In this research, the effects of modify bitumen with recycled waste plastic (RWP) were examined in terms of rheological, structural, chemical and performance characteristics of binder and asphalt mixture. The conclusions drawn from the work undertaken are as follow:

1. Six different types of RWPs were tested and, based on the melting point of these RWPs, only three RWPs were possible to be used for modifying the 160/220 grade penetration bitumen namely high density polyethylene (HDPE), low density polyethylene (LDPE) and polypropylene (PP).
2. The optimum blending time and temperature of RWPs namely LDPE, HDPE and PP modified binder were 120 minutes and 170^{o}C, respectively.
3. At the beginning, for each group of RWPs, sample were prepared by using the RWPs at 3%, 5%, 7% and 9% of bitumen weight. However, the blend containing 9% of RWPs could not be done because it appears to be fully saturated and reduce in workability. Hence, the 9% RWPs blends were discarded from the study.
4. Between the three different percentages of RWPs, 7% RWP give an improvement in performance properties. Thus, base binder containing 7% of RWP is chosen for further test.
5. Further comparison in terms of performance test has been made between 7% RWP-modified bitumens with another two conventional binders namely 40/60 penetration grade bitumen (written as control binder, CB) which represents the

similar penetration grade of high modification (7% RWP-modified binders) of base binder and a SBS-modified bitumen representing a ready-made polymer modified bitumen (PMB).

6. Based on the performance tests, the RWP-modified bitumen exhibited an improvement in rutting resistance and fatigue particularly at 20^{o}C.
7. Durability of bitumen was tested by means of Peel test in dry and wet condition (for 14 and 28 days). In dry condition, the SBS-modified bitumen showed higher fracture resistance compared to all binders tested. While among three modification level of RWP-modified bitumen, 7% exhibits higher fracture energy and thus, only 7% RWP-modified bitumens were used for evaluating the durability against moisture. In wet condition, the SBS-modified bitumen displayed good fracture resistance while among RWP-modified bitumens, the HDPE-modified bitumen has higher fracture resistance.
8. Among three types of RWP used, the HDPE presented the best performance in fatigue cracking and fracture properties while PP showed good rutting resistance.
9. Asphalt mixture performance tests were conducted for fatigue cracking at 20^{o}C and rutting at 40^{o}C. The binders used in asphalt mixture consists of conventional binders namely SBS-modified bitumen and 40/60 grade penetration bitumen (control binder) and study binders namely 7LDPE-modified bitumen, 7HDPE-modified bitumen and 7PP-modified bitumen. Two binder content were used which are 5% and 6% of weight of asphalt mixture.
10. For fatigue cracking test, better fatigue life was shown by asphalt mixture consisting 6% binder content in which the SBS-modified mixture has good performance in fatigue in comparison with all binders. However, all RWP-modified mixtures did not

exhibit better fatigue life and thus, not advisable to be used at lower temperature and slow moving vehicle road (lower speed road).

11. In term of rutting performance, the 6% bitumen content provide the lower rutting susceptibility. SBS-modified mixture showed the higher resistance to the permanent deformation. Nevertheless, the asphalt mixture containing HDPE and LDPE seem to be more pronounced on rutting resistance than control asphalt mixture and thus, these asphalt mixtures can be used at higher temperature and faster moving vehicle road (higher speed road). This indicates that the RWP-modified mixtures can perform better than control mixture that is having similar penetration grade of bitumen.
12. Although the RWP-modified bitumens did not perform as good as the commercial bitumen, its application can help to reduce the environmental pollution and at the same time can lead to possible cost saving for the industry.

Table 9.1: Binder suitability related to scenarios according to the asphalt mixture performance test

Condition	Asphalt Mixtures
Cold climates and slow moving vehicle (low speed road)	– SBS at 6% binder content
Hot climates and faster moving vehicle (higher speed road)	– SBS, HDPE and LDPE at 6% binder content

9.2 Recommendations

1. Some of the phase angle master curve of RWP-modified bitumens showed a discontinuous curve which may be caused by inappropriate geometry of the spindle used namely 8 mm and 25 mm. It is recommended to use spindle with geometry of 15 mm or torsion bar geometry and try to find out whether it makes the curve continuous or vice versa.
2. The rheological test is recommended to be done on the bitumen samples that have been moisturised after underwent peel test for evaluating the ageing effect during conditioning in details.
3. The type of aggregate and asphalt mixture design used in this study is limited to one type only. The effect of using different types of aggregate and asphalt mixture design is recommended for better understanding.
4. To gain more confidence in using RWP-modified bitumen, a comprehensive durability analysis could be done.
5. A study regarding the Life Cycle Cost Analysis (LCCA) of RWP-modified bitumen is recommended for evaluating its effectiveness based on cost and life cycle.
6. Further study should investigate the Life Cycle Assessment (LCA) analysis for measuring the cost and environmental impact of the RWP-modified bitumen.
7. The study on recyclability of RWP-modified bitumen will lead to better evaluation of this binder in term of the sustainable design.

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