THE EFFECT OF CRUMB RUBBER MODIFIER TO THE PROPERTIES AND RHEOLOGICAL BEHAVIOUR OF ASPHALT BINDER

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ABSTRACT

Several road pavement distresses are related to bitumen properties. Rutting and fatigue cracking are major distresses that lead to permanent failure in pavement construction.

The increased use of crumb rubber modifier in flexible pavements justifies the necessity for a better understanding of its physical and rheological characteristics as well as its elasticity behaviour. Basically the performance and properties of rubberised bitumen binder are influenced by the blending conditions and crumb rubber modifier content.

Thus, the main objective of this research is to investigate the effect of blending time, blending temperature and crumb rubber content on the performance properties of rubberised bitumen binders. In addition, this research aims to investigate the effect of crumb rubber modifier concentration on the rheological and physical properties of rubberised bitumen after short- term aging and long- term aging.

Laboratory investigation has been conducted on asphalt binder properties as a function of various blending temperatures, blending times and different crumb rubber modifier (CRM) percentages. Twenty CRM binder combinations (2 blending times, 2 blending temperatures and 5 CRM percentages by weight of binder) have been prepared as test samples and evaluated. Six different binder tests have been conducted. Namely (penetration test, Brookfield viscosity test, dynamic shear rheometer test (DSR), ductility test, elastic recovery test and softening point test).

In order to evaluate the durability performance of rubberised bitumen, aging tests have been conducted using short-term aging (i.e., rolling thin film oven test or RTFOT) and long-term aging (i.e., pressure aging vessel or PAV).

The results of this study showed that crumb rubber content and blending temperature for rubberised bitumen binders have a significant effect on modified binder properties while the blending time showed insignificant effect on modified bitumen binder properties in the case of 30 and 60 minutes. Higher blending temperature for rubberised bitumen binder resulted in an increase in viscosity, complex shear modulus, softening point, elastic recovery and softening point as well as a decrease in penetration, phase angle and ductility.

Statistical analysis using two-way ANOVA showed that CRM is statistically significant in terms of increasing penetration of asphalt binder, elasticity, viscosity, rutting factor $G^*/\sin(\delta)$ at 76 °C and resistance to flow.

Moreover, the results showed that the use of rubberised bitumen binder reduced the aging effect on physical and rheological properties of the bitumen binder as illustrated through lower aging index of viscosity, an increase in $G^*/\sin\delta$ at 76 °C, a decrease in phase angle (δ) with increased crumb rubber modifier content, indicating that the crumb rubber might improve the oxidative aging resistance of rubberised bitumen binder. In addition, the results show that the softening point increment (ΔS) and penetration aging ratio (PRA) of the rubberised bitumen binder decreased significantly due to crumb rubber modification. Furthermore, the higher crumb rubber content, the lower $G^*\sin\delta$ at 31 °C after PAV aging, thus leading to higher resistance to fatigue cracking.

ABSTRAK

Beberapa jenis kegagalan pada jalan raya berturap adalah berkaitan dengan sifat-sifat bitumen. Kegagalan jenis aluran dan tegasan berulang adalah punca utama yang menjurus kegagalan tetap dalam pembinaan jalan raya berturap. Peningkatan penggunaan serbuk getah dalam jalan raya berturap jenis mudah lentur menegaskan bahawa pemahaman yang tinggi tentang sifat-sifat fizikal dan reologi serta keanjalannya adalah satu keperluan. Pada asasnya, prestasi dan sifat-sifat penurapan getah berbitumen dipengaruhi oleh keadaan-keadaan pengadunan dan kandungan bahan tambah getah.

Oleh sebab itu, objektif utama penyelidikan ini adalah untuk menyiasat kesan pengadunan masa, suhu semasa proses pengadunan dan kandungan bahan tambah getah terhadap ciri-ciri penurapan jalan raya berbitumen dengan getah. Tambahan pula, penyelidikan ini juga menyiasat kesan fizikal dan reologi penggunaan getah sebagai bahan tambah dalam bitumen untuk jangka masa pendek dan jangka masa panjang (selepas pengoksidaan). Penyiasatan dalam makmal telah dijalankan ke atas sifat-sifat getah berbitumen (CRM) dengan menggunakan beberapa suhu pengadunan, masa pengaduann dan peratusan CRM yang berlainan. Terdapat dua puluh gabungan CRM (2 masa pengadunan, 2 suhu pengadunan dan 5 peratusan CRM berdasarkan berat bitumen) telah disediakan sebagai sampel-sampel ujian untuk dinilai dan diujikaji. Enam jenis ujian berbitumen telah dijalankan, iaitu (ujian penusukan, ujian kelikatan Brookfield, dinamik ricih reometer atau ujian DSR, ujian kemuluran, ujian pemulihan kenyal dan ujian takat lembut).

Penilaian prestasi ketahanan penurapan jalan berbitumen dengan getah, ujian dalam makmal telah dilakukan dengan menggunakan ujian penuaan jangka masa pendek (iaitu, ujian ‘ketuhar filem nipis beralun atau RTFOT) dan ujian penuaan jangka masa panjang (iaitu, menekan kapal lama atau PAV). Keputusan-keputusan kajian menunjukkan kandungan getah dalam bitumen dan suhu pengadunan mempunyai perbezaan yang nyata terhadap sifat-sifat getah berbitumen untuk penurapan jalan raya berbitumen dengan getah manakala masa pengadunan menunjukkan perbezaan yang nyata pada terhadap sifat-sifat getah berbitumen pada masa pengadunan 30minit dan 60minit. Getah berbitumrn pada suhu pengadunan yang lebih tinggi menyebabkan peningkatan dalam kelikatan, modulus ricih kompleks, takat lembut, pemulihan kenyal dan takat lembut tetapi penurunan dalam penembusan, sudut fasa dan kemuluran.

Analisis statistik menggunakan ANOVA dua hala menunjukkan perbezaan nyata pada CRM dari segi peningkatan nilai penusukan bitumen, keanjalan, kelikatan, faktor aluran G*/ sin (δ) pada 76 °C dan rintangan mengalir. Tambahan pula, keputusan menunjukkan penggunaan penuraparan jalan raya dengan getah boleh mengurangkan kesan penuaan terhadap sifat-sifat fizikal dan reologi bahan pengikat bitumen seperti yang digambarkan melalui kelikatan indeks penuaan yang lebih rendah, peningkatan dalam G*/ sin δ pada 76 ºC, satu pengurangan dalam ‘phase angle’ (δ) dengan pertambahan kandungan serbuk getah dalam bitumen, ia menunjukkan yang serbuk getah boleh meningkatkan rintangan penuaan pengoksidaan dalam penurapan dengan getah berbitumen. Sebagai tambahan, keputusan-keputusan juga menunjukkan pertambahan takat lembut (ΔS) dan nisbah penuaan penusukan (PRA) dalam penurapan dengan getah berbitumen menurun dengan banyaknya disebabkan pengubahsuaian getah berbitumen. Tambahan pula, semakin tinggi kandungan getah itu, lebih rendah G* δ di 31 ºC selepas penuaan melalui proses PAV, maka rintangan adalah lebih tinggi untuk penuaan dan tegasan berulang.

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Field of Study: Pavement materials

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My parents and my family for their love,
My beloved Husband for his endless help and continuous support,
My daughters,
Noor and Manar,
My sons,
Hassan and Hussein,
for their sweet smiles that have inspired me life and energy
towards my work.

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LIST OF ABBREVIATIONS AND SYMBOLS

ASTM: American Society of Testing Materials

AIRF: Aging index rutting factor (G*/sin δ)

AMWFA: Artificial marble waste fine aggregate

CRM : Crumb rubber modifier

DSR : Dynamic shear rheometer

FHWA Federal Highway Administration
G* : Complex shear modulus

G’ : Storage shear modulus

G’’ : Loss shear modulus

HMA : Hot mix Asphalt

min. : minute

n : number of samples

PAR : Penetration aging ratio

PAV ; Pressure aging vessel.

RTFOT : Rolling thin film oven test

SBR : Styrene Butadiene Rubber

SBS Styrene Butadiene Styrene

SHRP : Strategic Highway Research Program

VAI: Viscosity aging index

WCFA: Waste concrete fine aggregate

δ : Phase angle

ΔS : Softening point increment

CHAPTER 1

INTRODUCTION

1.1 Introduction

Bitumen is a visco-elastic material; its rheological properties are very sensitive to temperature and the rate of loading. Bituminous binders are used for road construction, pavement mixtures, surface dressing, bridge deck waterproofing, as joint and crack seal as they are durable and highly waterproof (Navarro *et al*., 2002).

Several road pavement distresses are related to bitumen properties. Rutting and fatigue cracking are among the major distresses that lead to permanent failure of the pavement surface. The rheological properties and durability of conventional bitumen however, are not sufficient to resist pavement distresses. In recent times, a serious problem that leads to environment pollution is the abundance and increase of waste tyre disposal. Large amounts of rubbers are used as tyres for cars and trucks etc. Despite the long run in service, these tyres are not discarded. Although, rubber as a polymer is a thermosetting material cross linked to processing and moulding, however it cannot be softened or remoulded by re-heating unlike other types of thermoplastics polymer which can be softened and reshaped when heated. The major approach to solve this issue is the recycle and reuse of waste tyre rubber, and the reclaim of rubber raw materials (Adhilkari *et al*., 2000).

Since the 1960s, the use of rubberised bitumen binder in road materials applications has gained increased interest in the paving industry. Hence, the task of current asphalt researchers and engineers is to look for different kinds of modified bitumen with rheological properties that would directly affect the asphalt pavement performance.

There are many modification processes and additives that are currently used in bitumen modifications such as Styrene Butadiene Styrene(SBS), Styrene-butadiene rubber(SBR), ethylene vinyl acetate (EVA) and crumb rubber modifier (CRM) . The use of commercial polymers such as SBS and SBR in road and pavement construction will increase the construction cost as they are highly expensive materials. However, with the use of alternative materials such as crumb rubber modifier (CRM), it will definitely be environmentally beneficial, and not only it can improve the bitumen binder properties and durability, but it also has a potential to be cost effective.

The improved property of rubberised bitumen is likely to depend on interaction between crumb rubber and bitumen binder. Crumb rubber particles swell when mixed with the bitumen binder to form a viscous gel; leading to an increase in the viscosity of the rubberised bitumen binder (Airey *et al*., 2003; Zanzotto and Kennepohl, 1996; Yildirim, 2007; Jeong *et al*., 2010; Bahia and Davis, 1994).

The properties of rubberised bitumen binders at a wide range of temperatures are highly dependent on the chemistry of the bitumen binder, the crumb rubber content, size and texture of rubber particle and the blending conditions (of time and temperature). Previous research has shown that the major mechanism of the bitumen – rubber interaction is the swelling of the rubber particles because of the absorption of the light fractions oil into the rubber particles and stiffening of the residual binder (Shen *et al*., 2009; Airey *et al*., 2003; Abdelrahman and Carpenter, 1999; Shen and Amirkhanian, 2005). Another study by Katman *et al*. (2005a; 2005b) using crumb rubber in porous asphalt showed that the blending procedure and mixing type also affect the performance of the rubberised porous asphalt.

In recent years, researches on applications of rubberised bitumen binders have reported many advantages. These advantages include improved bitumen resistance to rutting due to high viscosity, high softening point and better resilience, improved bitumen resistance to surface initiated cracks, reduction of fatigue/ reflection cracking, reduction of temperature susceptibility, improved durability as well as reduction in road pavement maintenance costs (Liu *et al*., 2009).

Bituminous binders are complex materials exhibiting elastic, visco-elastic and viscous behaviours under changing temperature and/or loading rate settings. The incorporation of tyre rubber into bitumen binder may enhance the properties of the base binder, yet it also adds complexity to the binder behaviour. Recently, crumb rubber modified bitumen binder testing has begun to draw more interest. Engineers have realised the potential of these materials and have begun to understand the relationships between laboratory analysis and field performance in a binder system.

1.2 Problem Statement

Over the years, road structures have deteriorated more rapidly due to increases in service traffic density, axle loading and low maintenance services. To minimise the damage of pavement surface and increase durability of flexible pavement, the conventional bitumen needs to be improved with regards to performance related properties, such as resistance to permanent deformation (rutting). The modification of bituminous binder has been explored over the past years in order to improve road pavement performance properties.

According to Ibrahim *et al*., (2009) Malaysia's production of scrap tyres is about 10 million pieces per annum and unfortunately they are being disposed in an environmentally unfriendly manner. A conventional bitumen 80/100 penetration grade is commonly used in Malaysia and moreover, it is subjected to high traffic loading and hot weather conditions. Thus, the use of crumb rubber in bitumen modification is considered as a sustainable technology which would transform unwanted residue into a new bituminous mixture which is highly resistant to rutting and fatigue deformations.

1.3 Study Aim and Objectives

Scrap tyres lead to serious disposal problems. However, the use of scrap tyres in asphalt pavements in the form of fillers/additives could minimise environmental pollution and maximise natural resource conservation. There are two major approaches to resolve the wastage of rubber and disposal of scrap tyres which are:

- To reduce and reuse used and waste rubber.
- To reclaim raw resource of rubber.

The aim of this study is to investigate the effect of adding crumb tyre rubber as additive to bitumen binder, using the wet process. The primary objectives are:

(i) To investigate the influence of blending condition mix of blending time (30 and 60 minutes) and blending temperature (160 and 180 °C) on the performance properties of control binder and modified bitumen binders.

(ii) To investigate the influence of various crumb rubber content (0%, 4%, 8%, 12%, 16% and 20%) on the performance properties of control binder and modified bitumen binders.

(iii) To investigate the effects of the rheological characteristics of crumb rubber bitumen binders on the permanent deformation properties.

(iv) To investigate the effects of aging methods (long term aging and short term aging) on the permanent deformation properties (rutting at high temperature).

1.4 Scope of Study

The scope of the study can be outlined as below:

1. Preparation of rubberised bitumen binder using five concentrations of crumb rubber (0%, 4%, 8%, 12%, 16% and 20%) respectively by binder weight.
2. Preparation of rubberised bitumen binder includes various blending condition of blending time (30 minutes and 60 minutes) and blending temperature (160 ºC and 180 ºC).
3. Testing the physical and rheological properties of all rubberised bitumen samples under different blending conditions and rubber content, by penetration test, softening point test, ductility test, elastic recovery test, brookfield viscosity test and dynamic shear rheometer test.
4. Testing the effect of rubber content and aging conditions on the rheological behaviour of rubberised bitumen after short term aging and long term aging. In addition, the study will include the investigation of crumb rubber content on rutting at high temperature and fatigue at intermediate temperature by testing both rutting factor G*/ sin (δ) at 76 ºC and fatigue factor G* sin (δ) at 31 ºC respectively.

1.5 Significance of Study

From the results of this study, the researcher aspires to consider crumb rubber modifier in hot mix asphalt to improve resistance to rutting and pavements with better durability by minimising the distresses caused in hot mix asphalt pavement. Hence, road users would be ensured of safer and smoother roads. Furthermore, the use of crumb rubber modifier as an additive in bitumen modified binder would reduce pollution problems and protect our environment as well.

1.6 Limitation of study

This study covers the topic of crumb rubber modified bitumen with different blending conditions for various crumb rubber content. In this study, the samples covered only two types of blending conditions that were 30 min and 60 min blending time and 160 °C and 180 °C blending temperatures. Perhaps, other categories of blending conditions such as more blending times, varying blending temperatures, different blending procedures and various mixing types could be considered for further studies in the future.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Roadways are considered as one of the most important elements of infrastructure and they play an essential role in our daily lives. In road pavement construction, the use of crumb rubber in the modification of bitumen binder is considered as a smart solution for sustainable development by reusing waste materials (Hamed, 2010). It is believed that crumb rubber modifier (CRM) could be one of the alternative polymer materials in improving bitumen binder performance properties of hot mix asphalt.

In this chapter, literature review on the use of crumb rubber in asphalt pavement will be presented and the factors that control the interaction of bitumen- rubber blends will be illustrated.

2.2 Bitumen Constitution

2.2.1 Definition of Bitumen

Bitumen is a dark black semisolid material, obtained from the atmospheric and vacuum distillation of crude oil during petroleum refining which is then subjected to various other processes (Croney and Croney, 1992). Also, it can be found as natural asphalt in nature’s surface deposits such as Trinidad Lake Asphalt (Read and Whiteoak, 2003).

It is considered as a thermoplastic visco-elstic adhesive and it has been used for road and highway pavement engineering, primarily because of its good cementing power and waterproof properties (Rozeveld *et al*., 1997). The analysis of bitumen indicates that the mix is approximately 8-11% hydrogen, 82-86% carbon, 0-2% oxygen, and 0-6%

sulphur by weight with rather small amounts of nitrogen, vanadium, nickel and iron (Rozeveld *et al*., 1997).

2.2.2 Bitumen as Colloidal System

Bitumen as a colloidal system consists of high molecular asphaltene/resin micelles diffused in an oily medium of lower molecular weight, constituted by naphthenic aromatics and saturated compounds. The micelles are mainly a composite of asphaltenes and resins acting as stabilisers to the mixture. The asphaltenes and resins may be related in an open network yet they can be disintegrated as discrete particles by varying the amounts of resin, asphaltene and oils in percentages (Rozeveld *et al*., 1997).

A study by Loeber *et al*. (1998) illustrated the rheological properties related to bitumen colloidal behaviour. Bitumen possesses a strong temperature dependence on rheological properties organised by the interaction of individual constitution (asphaltence, resins, aromatics, saturates). This study reported that an increase in one of these constitutions would change the structure and rheological behaviour of bitumen. Thus, bitumen with high asphaltence/resins ratio, would lead to a network structure with more rigidity and elasticity (low in phase angle and high in complex shear modulus), unlike the case of bitumen with high resins/asphaltence ratio which leads to high viscous behaviour.

Figure 2.1 shows the representative structures of four generic groups (SARA): saturated, aromatic, resins (which form the maltene fraction) and asphaltenes. This model is based on the colloidal (micellular) model (Masson *et al*., 2001; Nellensteyn, 1924). The complexity, content of heteroatom, aromatic and increase of molecular weight are in the order of S< A< R< A (Claudy *et al*., 1991).

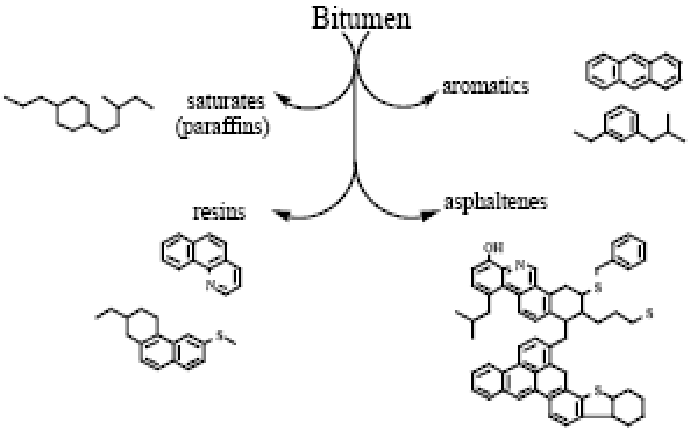


Figure 2.1 Representative structures of bitumen fractions: saturates, aromatics, resins and asphaltenes (SARA) (Masson *et al*., 2001)

Resins are an intermediate weight, semi-solid fraction formed of aromatic rings with side chains. Also, resins are polar molecules that act as peptising agents to prevent asphaltene molecules from coagulating. The lightest molecular weight materials are the non-polar oils. Oils generally have a high proportion of chains as compared to the number of rings. From literature, the resins and oils are referred to collectively as maltenes. In general, asphaltenes produce the bulk of the bitumen while resins contribute to adhesion and ductility and oils influence flow and viscosity properties (Krebs and Walker, 1971).

In addition, the component of bitumen can be recognised as asphaltenes and maltenes. The viscous-elastic properties of bitumen and its properties as a paving binder are determined by the differing percentages between asphaltenes and maltenes fraction (Navarro *et al*., 2002; Lewandoweski, 1994; Dongre *et al*., 1996). According to the microstructure and the colloidal system, asphaltenes are diffused into an oily matrix of maltenes, an encased by a shell of resins whereby its thickness varies with the temperature that is being tested (Navarro *et al*., 2009).

Thus, bitumen composition and temperatures are strongly dependent on the mechanical properties, microstructure of bitumen and on the degree of aromatisation of the maltenes and the concentration of asphaltenes (Widyatmoko and Elliott ,2008; Navarro *et al*., 2009).

2.2.3 Bitumen Polarity

Bitumen has another important property which is polarity, which is the separation of charge within a molecule. Polarity is an important factor in the bitumen system because it refers to molecules managing themselves into preferred orientations.

According to Robertson (1991), most of the naturally occurring heteroatoms, nitrogen, sulfur, oxygen and metals are strongly dependent on polarity within these molecules. Also, oxidation products form upon aging are polar and further contribute to the polarity of the entire system. The physicochemical properties have an obvious significant effect on bitumen and each reflects the nature of the crude oil used to prepare it.

According to Robertson (1991) the most consistent description, or model, of petroleum bitumen polarity is as follows. Bitumen is a collection of polar and non-polar molecules:

- The polar molecules are associated strongly to form organised structures and represent a more stable thermodynamic state.

- The non-polar model has the ability to dissociate the organised structure, but again there are possible variations from bitumen sources and its viscous behaviours are highly dependent on the temperature.

2.2.4 Bitumen Rheology

Rheology is the study of deformation and flow of materials. It is the science knowledge that is related to all aspects of deformation of material under the influence of external stresses (Ferguson and Kemblowski, 1991).

Bitumen behaves in a unique manner depending upon both the load applied and the rate of loading. Temperature additionally is a factor that could be correlated with the rate of loading. At elevated temperatures, or slow rates of loading, bitumen becomes a viscous material. However, at decreased temperatures or higher rates of loading, bitumen then becomes a highly elastic material. In fact at intermediate temperatures, bitumen has two different characteristics; i.e. an elastic solid and a viscous fluid (Van der Poel, 1954). The study of bitumen rheology is an important phenomenon to characterise the dynamic mechanical behaviour of binders.

Bitumen binders are referred to as visco-elastic materials because they exhibit combined behaviour (properties) of elastic and viscous material as presented in Figure 2.2 (a) with the removal of the applied stress from the material, there is a complete recovery to the original position. Figure 2.2(b) explains the behaviour of a viscous material in case the strain of the material increases through time under stable stress. Figure 2.2(c) illustrates the behaviour of a visco-elastic material when stable stress increases the strain over a long period of time and when the applied stress is removed, the material loses its ability in attaining its original position resulting in permanent deformation (Van der Poel, 1954).

According to Van der Poel, (1954), generally the stiffness modulus of bitumen binders can be defined by:

$$S(t) = \sigma/\varepsilon(t) \qquad \text{Eq. (2.1)}$$

Where:

- S (t) = dependent stiffness modulus (Pa)
- t = loading time (s)
- σ = applied constant uniaxial stress (Pa)
- ε(t) = uniaxial strain at time t, (m/m)

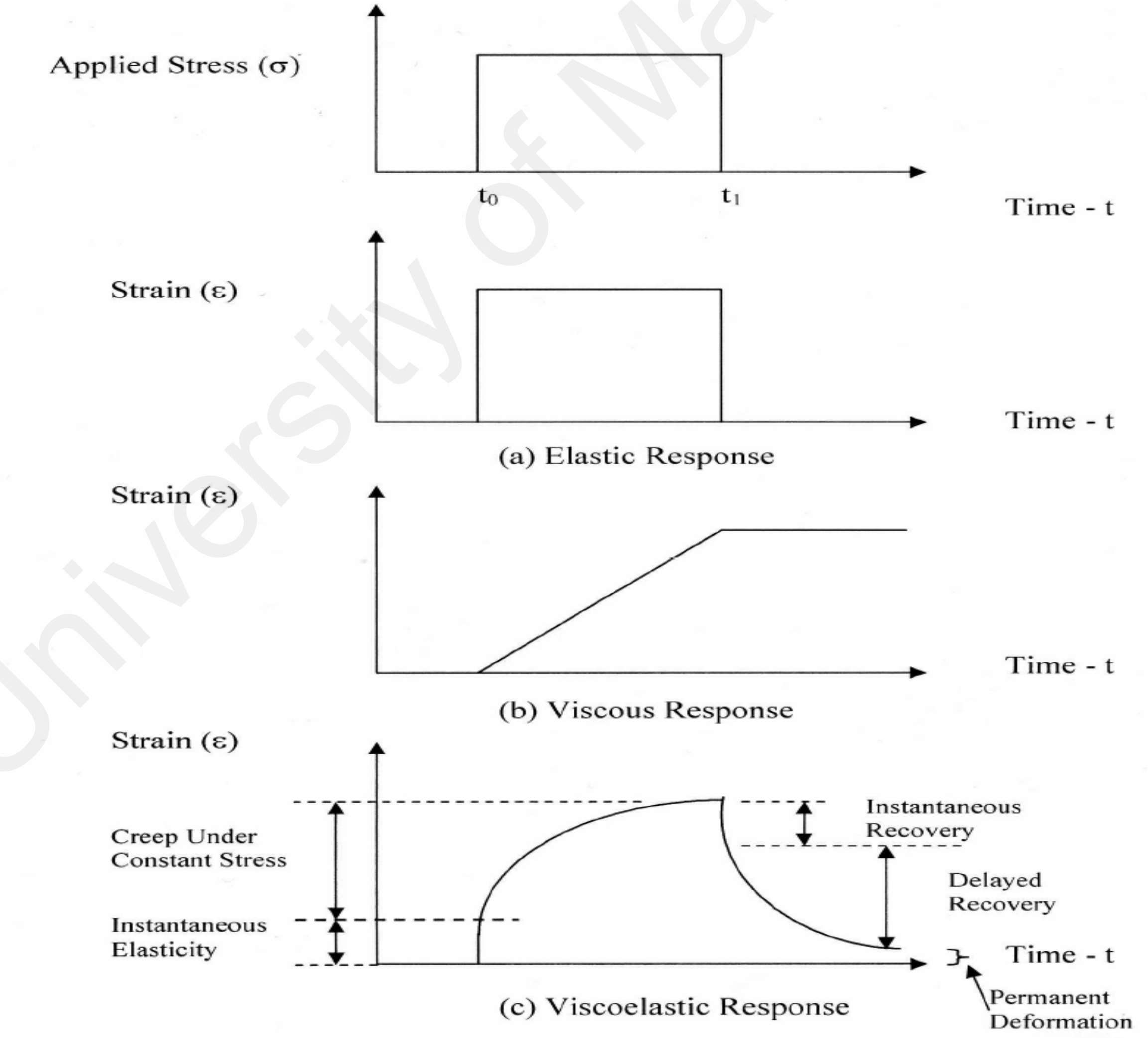


Figure 2.2 General behaviour of elastic, viscous and viscoelastic material under constant stress loading (Van der Poel, 1954).

2.3 Bitumen Ageing and Physical Hardening Properties

The factor influencing the performance and characteristics of bitumen binder is the phenomenon known as aging. Many factors might contribute to this hardening of the bitumen such as oxidation, volatilisation, polymerisation and thixotropy. This is because bitumen is an organic compound, capable of reacting with oxygen found in the environment. The bitumen composite changes with the reaction of oxidation developing a rather brittle structure. This reaction is referred to as age hardening or oxidative hardening (Peterson, 1984).

Volatilisation occurs when the lighter components of the bitumen evaporate. In general, this is related to elevated temperatures that are found firstly during the hot mix asphalt production process. Polymerisation is the means by which resins are assumed to combine into asphaltenes, resulting in an increase in the brittleness of the bitumen along with a tendency toward non-Newtonian behaviour. At the end of the reaction, thixotropy, or an increase in viscosity over time, also contributes to the aging phenomenon in bitumen (Peterson, 1984).

However, the most important factors in the aging process of bitumen binder seem to be oxidation and volatilisation. The occurrence of steric hardening and the time-dependent reversible molecular association have affected the binder properties but this is not considered as aging. Steric hardening is only a factor at intermediate temperatures; at high temperatures excess kinetic energy in the system prevents the association and at low temperatures the rate of association is found to be slower due to the binder's high viscosity (Peterson, 1984).

Bahia and Anderson (1993) studied the mechanism by which binder properties may change at low temperature. This mechanism is called physical hardening occurs at temperatures next to or lower than the glass transition temperature and causes significant hardening of the bitumen binder.

The rate and magnitude of the hardening phenomena has been observed to increase with decreasing temperatures and is reported to be similar to the phenomena called physical aging on amorphous solids (Struik, 1978).

The physical hardening can be explained using the free volume theory which introduced the relationship between temperature and molecular mobility. The free volume theory includes the molecular mobility dependent on the equivalent volume of molecules present per unit of free space or free volume. Based on the free volume theory, when amorphous material is cooled from a temperature above its glass transition temperature, molecular adjustments and the collapse of free volume rapidly show a drop in temperature. At that temperature, the structural state of the material is frozen-in and deviates from thermal equilibrium due to the continuous drop in kinetic energy. Hence, it has been postulated in order for physical hardening to happen in binders, temperatures must be higher than the glass transition temperature (Marasteanu and Anderson, 1999; Doolittle 1951).

2.4 Performance Grading (PG) of Bitumen Materials

The inability of conventional methods to correlate binder properties with pavement performance has led to the development of a new specification called Superpave (Superior Performing Asphalt Pavements) performance grade in the early 1990's.

Hence, the Superpave performance grading system appears to be a better tool than conventional methods since binder properties and behaviour are being compared at the conditions under which they are being used. This performance grading (PG) system attempts to properly classify asphalt binders based on their performance (Strategic Highway Research Program, 1994).

A number of current studies have shown that binders of the same performance grading based on Superpave specification (PG grades) tend to show serious differences with regards to their performance at low temperatures. This approach is specifically to enhance pavement performance whereby pavement distresses can be reduced under various climatic conditions through an appropriate choice of appropriately modified bitumen binders (McGennis, 1995).

2.5 Characteristics of Bitumen Binder Modification

The complexity in the chemistry of bitumen products is due primarily to the intricate composite of petroleum crude oils which bitumen products are derived. Due to the inherent weaknesses of conventional bitumen which have led high maintenance cost of the highway systems, there has been need to modify bitumen. Modification/reinforcement of bitumen binder is possible during different stages of its usage, either in between binder production and mix processes or before paving mix production (Richard and Bent, 2004).

According to Larsen *et al.* (1988) the bitumen modification provides binders with:

- Sufficient increase in consistency at the highest temperature in pavements to prevent plastic deformation.

- An increase in flexibility and elasticity of binders at low temperature to avoid crack deformations and loss of chippings.
- An improvement of adhesion to the bitumen into aggregates.
- Improved homogeneity, high thermo stability and aging resistance helps reduce the hardening and initial aging of the binders during mixing and construction.

From the scientific point of view, the pavement layer should be capable of placement and compaction to provide strong riding surface and appropriate mix density. Bitumen binder together with mixing design must be able to withstand loading to avoid permanent deformation, repeated load and unloading without exhibiting fatigue failure.

2.6 Types of Additives for Bitumen Modification

The increased demand on highway roads might reduce its strength properties and make roads more susceptible to permanent distresses and failure. In general, pavement performance properties are affected by the bitumen binder properties; it is known that the conventional bitumen has a limited range of rheological properties and durability that are not sufficient enough to resist pavement distresses. Therefore, bitumen researchers and engineers are looking for different types of bitumen modifiers with excellent rheological properties, which directly affect asphalt pavement performance. Over several years, there have been various types of additives that have been used as modified agents into bitumen under specific conditions of mix, such as SBS, SBR, natural rubber and crumb tyre rubber.

2.6.1 Styrene Butadiene Styrene (SBS)

Styrene- butadiene- styrene (SBS) is a block copolymer which improves the elasticity of asphalt cement (Yildrim, 2007). SBS is defined as a thermoplastic rubber with very strong and elastic behaviour. Moreover, these properties are derived from the cross linking of a three dimensional network counting on polymer element, the source of its strength presented by polybutadiene component while the styrene is the main source of the elasticity (Murphy *et al*., 2000).

According to Murphy *et al*. (2000), SBS was used to evaluate the properties of the bitumen 200 penetration grade (Middle East sourced). The results showed significant effects of SBS polymer on softening point and viscosity of modified bitumen binder compared to unmodified bitumen under the same conditions of tests.

Navarro *et al*. (2009) in their study compared the modification capability of two different types of bitumen modifiers: non reactive polymer which included a block copolymer (SBS) at a processing temperature of 180 ºC and a reactive polymer (diphenylmethane diisocyanate polyethylene glycol prepolymer MDI–PEG) at a processing temperature of 90 ºC. The results showed that the non reactive polymer (SBS) should be used in high concentration to achieve better results for bitumen modification. Thus, the use of this copolymer in bitumen modification is not only costly but less appropriate in pavement constructions and maintenances works.

The improvement of polymer modifier asphalt mixture with SBS, polyalphaolefin, cellulose fibre and in relation to the results of LCPC(Laboratoire Central des Ponts et Chaussées) wheel tracking and repeated creep tests, SBS mixtures were evidently the most significant mixtures in tandem with resistance to rutting (Tayfur *et al.,* 2007).

2.6.2 Synthetic Rubber- Styrene- Butadiene (SBR)

Styrene-butadiene rubber (SBR) is an elastomeric polymer modifier and an example of a random copolymer, formed by a random reaction of styrene and butadiene together.

The behaviour of SBR is between the flexibility of butadiene and the stiffness of styrene as well as a function of the relative proportions of the two components. Random copolymers act as homogeneous systems that exhibit single-phase morphology and do not create a network. In addition, they demonstrate distinct characteristics between the extremes of the reacting monomers (Noshay and McGrath, 1977).

Ruan *et al*. (2003) conducted a study on the effect of long term aging on rheological properties of polymer modified binder. In this study, SBR, SBS and tyre rubber were utilised as modifiers. From the results, the polymer modification resulted in increased complex shear modulus at elevated temperatures and vice versa with decreased temperatures as well as improved ductility. The bitumen temperature susceptibility decreased with oxidative aging. Apart from that, oxidative aging damaged the polymer network in binders, increased the relaxation spectrum and showed decreased polymer effectiveness in improving bitumen ductility.

Hwang and Ko (2008) did a comparative study of mechanical and physical properties of SBR-polymer modified mortars using recycled waste materials. The study centered on polymer modified mortars using either recycled waste concrete fine aggregate (WCFA) or artificial marble waste fine aggregate (AMWFA) which were prepared and examined with the aim of studying the feasibility of recycling. The results indicated that in the presence of SBR, the compressive strength decreased compared to the omission of SBR.

2.6.3 Natural Rubber

Natural rubber can be defined as an elastic hydrocarbon polymer that was originally found in nature, in the form of latex.

Tuntiworawit *et al.* (2005) studied the modification of asphalt using natural rubber in the form of latex. The main objective was to select the engineering properties of asphalt cement and asphalt concrete mixture treated with natural rubber latex at varying mix. The finding of the study showed that using natural rubber latex as an additive is an advantageous alternative for bitumen modification. Additionally, natural rubber as an additive was found to improve the performance of asphalt pavement in terms of flexibility and stability. The study selected a latex content of 9 % by weight of asphalt.

2.6.4 Crumb Tyre Rubber

Recycled waste tyre rubber is used as reclaimed rubber and referred to as crumb rubber (see Figure 2.3). Tyre rubber is a blend of synthetic rubber, natural rubber, carbon black, anti-oxidants, fillers and extender type of oils which are soluble in hot paving grade. Scrap rubber, crumb rubber and reclaimed rubber are various terms used to describe recycled rubber. Car and truck tyres are the largest recycled rubber sources. Car tyres are primarily made of Styrene Butadiene Rubber (SBR) or polyisoprene and carbon black. Truck tyres on the other hand, consist of a higher percentage of natural rubber compared to car tyres up to 30% of the combined polymer content (Becker *et al*., 2001; Caltrans, 2003).

Rubberised bitumen binders are obtained by the incorporation of crumb rubber from ground tyres in bitumen binder under certain conditions of time and temperature. There are three different processes involved to produce bitumen- rubber: the wet process, dry process and terminal blending (Takallou and Takallou, 2003).

Figure 2.3 Discarded waste tyres

Various blends of rubberised bitumen produced using both dry and wet processes are considered as good alternatives to conventional bitumen and hence, this has been investigated and evaluated thoroughly. Heating of bitumen- rubber mix during the curing processes induces some breaks in rubber crosses, as evidenced in devulcanisation (Billiter *et al*., 1997b).

This phenomenon induces a continuous reduction in the viscosity in the form of initial degradation of polymer chains, thus, swelling and devulcanisation occur during rubberised bitumen production. Nevertheless, the predominance depends greatly on processing parameters and generally on blending temperature and blending duration (Billiter *et al*., 1997b). The properties of rubberised bitumen binders produced in a wet process depend on the crumb rubber and bitumen binder used in the process (Dantas Neto, 2004).

2.7 History of Using Crumb Rubber as a Modifier in Bituminous Pavement

The earliest experiments date back to the 1840s, which involved incorporating natural rubber into bitumen to increase its engineering performance properties. The process of bitumen modification involving natural and synthetic rubber was introduced as early as 1843 (Thompson, 1979). Then, in 1923, natural and synthetic rubber modifications in bitumen were further improved (Isacsson and Lu 1999; and Yildrim, 2007). According to Yildrim, (2007) the development of rubber-bitumen materials being used as joint sealers, patches and membranes began in the late 1930s. In 1950, the use of scrap tyre in asphalt pavement was reported (Hanson *et al*., 1994).

In 1960, Charlie Mac Donald working as head material engineer in Phoenix, Arizona, used ground tyre rubber as an additive in bitumen binder modification. He found that after completing the mixing of crumb rubber with the conventional bitumen and allowing it to blend for mix duration of 45 – 60 minutes, there were new material properties produced, which resulted swelling in the size of the rubber particles at higher temperatures allowing for higher concentrations of liquid bitumen contents in pavement mixes (Huffman, 1980). In the mid-1980s, the Europeans began developing newer polymers and additives for use in bitumen binder modification (Brule, 1996).

In recent years, the use of crumb rubber has gained interested in pavement modification and has shown that crumb tyre rubber can improve the bitumen performance properties (Brown *et al*., 1997; Maupin, 1996; Charania *et al*., 1991; Stroup- Gardiner *et al*., 1996).

2.7.1 History of Using Crumb Rubber Modifier in Malaysia

In Malaysia, the use of rubber as an additive for road pavement construction supposedly started in the 1940s, but there has not been any official record of such practices. The first recorded trial using rubberised bitumen technology was reported in 1988, the wet mix process was used with the mix of rubber additives in the form of latex into bitumen binder (Sufian and Mustafa, 1997). In 1993, another rubberised road trial using waste gloves and natural rubber latex was carried out in Negeri Sembilan (Samsuri *et al*., 1995).

2.8 Crumb Tyre Rubber Grinding Process

Crumb rubber is made by shredding scrap tyre that is a particular material free of fibre and steel. The rubber particle is graded and found in many sizes and shapes. Crumb rubber is described or measured by the mesh screen or sieve size through which it passes during the production process. To produce crumb rubber, generally it is important to reduce the size of the tyres. There are two techniques to produce crumb rubber: ambient grinding and the cryogenic process (Becker *et al*., 2001)**.**

Ambient grinding process can be divided into two methods: granulation and cracker mills. Ambient describes the temperature when the waste tyres rubber as its size is reduced. The material is loaded inside the crack mill or granulator at ambient temperature. Cryogenic grinding is a cleaner, slightly faster operation resulting in production of fine mesh size. The high cost of this process is a disadvantage due to the added cost of liquid nitrogen.

2.9 Performance of Crumb Tyre Rubber in Bituminous Materials

There are two rather different methods in the use of tyre rubber in bitumen binders; first, is by dissolving crumb rubber in the bitumen as binder modifier. Second, is by substituting a portion of fine aggregates with ground rubber that does not completely react with bitumen (Huang *et al.,* 2007).

Numerous factors can influence the modification effects which include the base bitumen constituents , blending time and temperature, percentage of rubber, gradation of crumb rubber, type of mixing (wet or dry) and the grinding process method (Huang *et al.,* 2007; Airey *et al*., 2003; Jeong *et al*., 2010). It was observed that during the bitumen-rubber blending, due to higher stiffness and tensile strength at elevated temperatures, the mixture had decreased rutting capability (Palit *et al*., 2004).

The design method for conventional bitumen mixture can be used for bitumen-rubber mixture as the mix stability being the primary factor. Also, standard paving machinery can be used for placement of bitumen-rubber mixture. However, a pneumatic tyre roller is not suitable as asphalt rubber will stick onto the roller tyres (Huang *et al.,* 2007; Epps, 1994).

Rubber Pavement Association found that using tyre rubber in open- graded mixture binder could decrease tyre noise by approximately 50%. In addition, in spray applications, rubber particles of multiple sizes had a better sound absorbing (Zhu and Carlson, 2001). Moreover, another advantage of using asphalt rubber is to increase the life- span of the pavement. However, recommendations were made to assess the cost effectiveness of asphalt rubber (Huang *et al.,* 2007).

The benefits of using crumb rubber modified bitumen are as listed below:

- Lower susceptibility to varying temperature on a daily/ seasonal basis.
- More resistance to deformation at higher pavement temperature.
- Improved age resistance properties.

- Higher fatigue life for mixes, and better adhesion between aggregate and binder.

2.10 Physical and Rheological Properties of Rubberised Bitumen

2.10.1 Penetration Properties

Penetration is a measure of hardness or softness of bitumen binder which shows an effect by adding crumb rubber to bitumen binder; it decreases as rubber content is increased.

The penetration shows lower values as rubber content increases at different mix conditions of rubberised bitumen binder, indicating that the binder becomes stiff and more viscous (Katman 2006).

Mahrez (1999) investigated the properties of rubberised bitumen prepared by physical blending of bitumen 80/100 penetration grade with different crumb rubber content and various aging phases. The results of penetration values decreased over the aging as well as before aging by increasing the rubber content in the mix. Also, the modified binders have lower penetration values than unmodified binders.

Another study by Kumar *et al*. (2009) on penetration change was conducted using bitumen 80/100 and 70/100 penetration grade mixes with different crumb rubber percentage. The results showed a significant decrease in the penetration values of modified binder due to high crumb rubber content in the binders**.**

2.10. 2 Elastic Recovery Properties

Elastic recovery or elasticity describes the ability of a bitumen binder to elongate when tension is applied and to recover its original shape when the tension is released. The degree of elastic recovery was used as an indicator of permanent deformation in pavement materials (Yildirim, 2007; Shenoy, 2008).

According to Jensen and Abdelrahman (2006), elastic recovery property is very important in both fatigue and rutting resistance selection and evaluation. Elastic recovery is a property that indicates the quality of polymer components in bitumen binders. Oliver (1981) concluded from his study, that the elastic recovery of rubberised bitumen binders leads to an increase as the rubber particle size decreases.

Modified bitumen binders showed a significant enhancement on the elastic recovery, and, in contrast, the ductility decreased with respect to unmodified binders (Martinez *et al*., 2006).

2.10.3 Ductility Properties

Ductility is a distinct strength of bitumen, allowing it to undergo notable deformation or elongation. Ductility is defined as the distance in cm, to which a standard sample or briquette of the material will be elongated without breaking.

The effect of rubber type and its concentration on the properties of rubberised bitumen binders was evaluated. It was found that rubber types could affect the force ductility properties at 4 ºC (Rosner and Chehovits, 1982). They found that the ductility test at 4 ºC decreased with increase in rubber concentration and it was found that the load failure increased and the elongation at failure decreased as rubber concentration increased.

Another study by Frobel *et al*. (1978) concluded that finer rubber particles resulted in higher ductility elongation than larger particles size, and also that toughness would increase as particle size decreases. A combined effect of both time and temperature was noted with minimum elastic recovery value improved at maximum time and maximum temperature of two hours and 240 ºC respectively (Jensen and Abdelrahman, 2006).

Bitumen-rubber modification resulted in a better rutting resistance and higher ductility. However, the modified binder was susceptible to decomposition and oxygen absorption. There were problems of low compatibility because of the high molecular weight. Furthermore, recycled tyre rubber decreases reflective cracking, which in turn increases durability. In using waste tyre rubber there are however, several practical and experimental issues such as it requires an elevated composite of temperatures and extended digestion time during the mixing process for it to be diffused in the bitumen (Yildrim, 2007).

2.10.4 Viscosity and Softening Point Properties

Viscosity refers to the fluid property of the bitumen and it is a gauge of flow-resistance. At the application temperature, viscosity greatly influences the potential of the resulting paving mixes. During compaction or mixing, low viscosity has been observed to resulting in lower stability values. Softening point refers to the temperature at which the bitumen attains a particular degree of softening.

The use of crumb rubber in bitumen modification leads to an increase in the softening point and viscosity as rubber crumb content increases (Mahrez, 1999; Katman, 2006).

Mahrez and Rehan (2003) claimed that there is a consistent relationship between viscosity and softening point at different aging phases of rubberised bitumen binder. Also, it is reported that the higher crumb rubber content leads to higher viscosity and softening point.

Viscosity is a continuously increasing non linear function of rubber content and the relative increase is a factor related to the application of temperature (Bahia and Davies, 1995b).

2.11 Bitumen- Rubber Blends Interaction and Absorption

Modified bitumen using crumb rubber showed an improvement in the performance of pavements over the base binders as a result of the interaction of crumb rubber with base binders. Due to this interaction, there are noticeable changes in the viscosity, physical and rheological properties of the rubberised bitumen binder (Airey *et al*., 2002; Bahia and Davies, 1994; Lougheed and Papagiannakis, 1996; Oliver, 1983), leading to high resistance of rutting of pavements (Huang *et al*., 2002; Rebala and Estakhri, 1995).

According to Jensen and Abdelrahman (2006) there are three stages of interaction that have been evaluated with regard to bitumen - rubber binder:

- An early stage that occurs immediately after mixing crumb rubber with bitumen;

- An intermediate storage stage, during which the binder is held at elevated temperatures for up to a few hours before mixing with aggregate.
- An extended (storage) stage when bitumen- rubber blends are stored for extended periods before mixing with aggregate.

Miknis and Michon (1997) investigated the application of nuclear magnetic resonance imaging to rubberised bitumen binder. The application of this technology led to investigating the different interaction between crumb rubber and bitumen such as swelling by bitumen molecules, possible dissolution of rubber components in bitumen and devolatitisetion and crosskicking in rubber. The outcome of this study is swelling of rubber particles may depended on asphalt molecules.

Gaulliard and Leblanc (2004) using a torque rheometer investigated the swelling kinetics of vulcanised rubber particles. They designed a mathematical model which presented the physics involved in the processing of solvent uptake by rubber particles.

From the above review of literature, in summary, the primary mechanism of the interaction is swelling of the rubber particles caused by the absorption of light fractions into these particles and stiffening of the residual binder phase (Shen *et al.*, 2009; Abedlrahman and Carpenter, 1999; Airey *et al.*, 2003; Shen and Amirkhanian, 2005). The rubber particles are constricted in their movement into the binder matrix to move about due to the swelling process which limits the free space between the rubber particles. Compared to the coarser particles, the finer particles swell easily thus developing higher binder modification (Shen *et al.*, 2009; Abedlrahman and Carpenter, 1999).

The swelling capacity of rubber particle is linked to the penetration grade of the binder, crude source and the nature of the crumb rubber modifier (Airey *et al*., 2003). According to Shen *et al*. (2009) the factors which affect the digestion process of the bitumen and rubbers blends are as presented below:

- Rubber content
- Rubber size (gradation).
- Original binder viscosity.
- Blending conditions (blending temperature and blending time).

2.11.1 Rubber Content

According to a study conducted by Lee *et al*. (2008), the higher crumb rubber content produced increased viscosity at 135˚C and improved the rutting properties. It was also observed that the increased crumb rubber amount (fine crumb rubber) produced rubberised bitumen with higher viscosity and lower resilience (Dantas Neto, 2004).

However, optimum crumb rubber content still needs to be determined for each crumb rubber size and asphalt binder. It is believed that a physicochemical interaction occurs between the asphalt and the crumb rubber alters the effective size and physical properties of the rubber particle, thus influencing pavement performance (Huang, 2008).

Regarding the low temperature performance, an investigation with 18- 22% of rubber content, showed change that was little significance within this range in affecting the tensile and fracture performance of the bitumen compared to varying the binder content between 6-9 % by bitumen weight (Huang *et al*., 2007; Hossain *et al*., 1999).

A study by Khalid (2005) found that higher binder content led to longer fatigue life of rubberised bitumen mixture, better resistance to rutting as well as results showing good resistance to fracture and fatigue cracking. Liu *et al*. (2009) found that content of crumb rubber as the most significant affecting factor followed by crumb rubber type and lastly the size of the particle.

Becker *et al*. (2001) claimed that blend properties will be influenced by the amount of crumb rubber added to the bitumen. Higher amounts indicated significant changes in the blend properties. As rubber content generally increases, it leads to:

- Increased viscosity.
- Increased resilience.
- Increased softening point.
- Decreases penetration at 25˚C.

2.11.2 Rubber Particle Size

A study by Sousa and Weissman (1994) using a binder with 15% rubber content (size of 0.2, 0.4, 0.6 mm) in dense-graded bitumen. The mixture showed improved performance in dynamic stability, 48h residual stability, flexural strength and strain value. Asphalt containing 0.2 and 0.4 mm size rubber indicated the best laboratory results (Souza and Weissman, 1994). The particles size disruption of crumb rubber influenced the physical properties of bitumen- rubber blend. In general, small difference in the particles size has no significant effects on blend properties. However, the crumb rubber size can certainly make a big difference.

According to a study by Shen *et al*. (2009), the particle size effects of CRM on high temperature properties of rubberised bitumen binders was an influential factor on visco-elastic properties. Coarser rubber produced a modified binder with high shear modules and an increased content of the crumb rubber decreased the creep stiffness which in turn showed significant thermal cracking resistance.

2.11.3 Rubberised Bitumen Viscosity

The Asphalt Institute reported that viscosity of bitumen binders at high temperatures is an important property as it indicates a binder’s ability to be pumped through bitumen plant as well as coating the aggregate in hot mix asphalt mixture (HMA) (Asphalt Institute, 2003 ; Lee *et al*., 2008).

During the blending operation, bitumen and rubber can be adjusted, by diluting the petroleum distillates or aromatic extender oils with the blend. However, this may not be essential for low rubber content. The quantity and type of oil to be included varies depending on rubber size, quantity, bitumen source and grade, and reactions. Aromatic oils are evidently suitable for adjusting viscosity include Sundex, Duterx and Califlux. Another alternative approach for lowering the viscosity is by using a lower viscosity grade instead of extender oil. This however could result in a permanent reduction in viscosity (Shen *et al*., 2009).

A study by Thodsen *et al*. (2009) indicated that processing procedure and tyre type play an important role in the determination of rubberised bitumen viscosity. Interaction between crumb rubber and bitumen binders is referred to as a physical interaction where the crumb rubber through diffusion, absorbs the aromatic fraction of the bitumen binders which leads to swelling of the crumb rubber particles. This particle swelling compounded with reduction in the oily fraction of the binder results in increased viscosity in the rubberised bitumen binder.

Binder viscosity is vital for the pump ability of the binder and the workability of the bitumen mixture, but it is not directly related to the in service performance of the binder within a bitumen mixture. Also, due to the non-Newtonian characteristics of most particulate filled liquids, such as rubberised bitumen binder, viscosity measurements on its own may not to be an adequate precision in the description of complexities of the properties (Wypych, 2000).

2.11.4 Blending Conditions (Blending Temperature and Blending Time)

In general, the bitumen binder and ground tyre rubber are mixed together and blended at elevated temperatures for differing periods of time prior to using them as a paving binder. These two factors work together to evaluate the performance properties of rubberised bitumen binder through blending process of bitumen – rubber interaction. This variation in mixing time and temperature results due to the normal activities are related to bitumen paving construction (Shakor, 1997). Nevertheless, the consistency of rubberised bitumen rubber can be affected by the time and temperature used to combine the components and thus must be cautiously used for its optimum potential to be achieved.

Moreno *et al.* (2011) reported that the reaction time has no significant effect on the selection of the optimal binder content or on the compaction of the mixture. They mentioned that the digestion time/ interaction time have an impact on the mechanical performance of the mix.

Another study by Xiao *et al.* (2006) on the effects of reaction time on the permanent changes of crumb rubber after mixing with the bitumen showed that the reduction in size of rubber material increased with the blend duration and with decreasing size of crumb rubber.

According to Lee *et al.* (2006), who conducted a study on the evaluation of the aging difference between rubberised bitumen binder and control by using seven reaction times (5, 30, 60, 90, 120, 240 and 480 minutes) at the reaction temperature of 177 °C, concluded that a longer reaction time was ineffective in increasing the high temperature viscosity of the control binder.

On the whole, longer reaction time for production of the rubber modified binder apparently caused increased viscosity due to the increased rubber mass through binder absorption. DSR analysis displayed that the reaction time ranging from 0 to 480 minutes hardly has any effect on the increased high failure temperature in the control binder. However, DSR analysis also showed that the reaction times of 60, 90, 120, 240 and 480 minutes did not reflect a significant effect on high failure temperature in the rubber-modified binder. In addition, there was no difference in the molecular size variation between the control binder and the rubberised bitumen binders irrespective of the reaction time (Lee *et al.*, 2006).

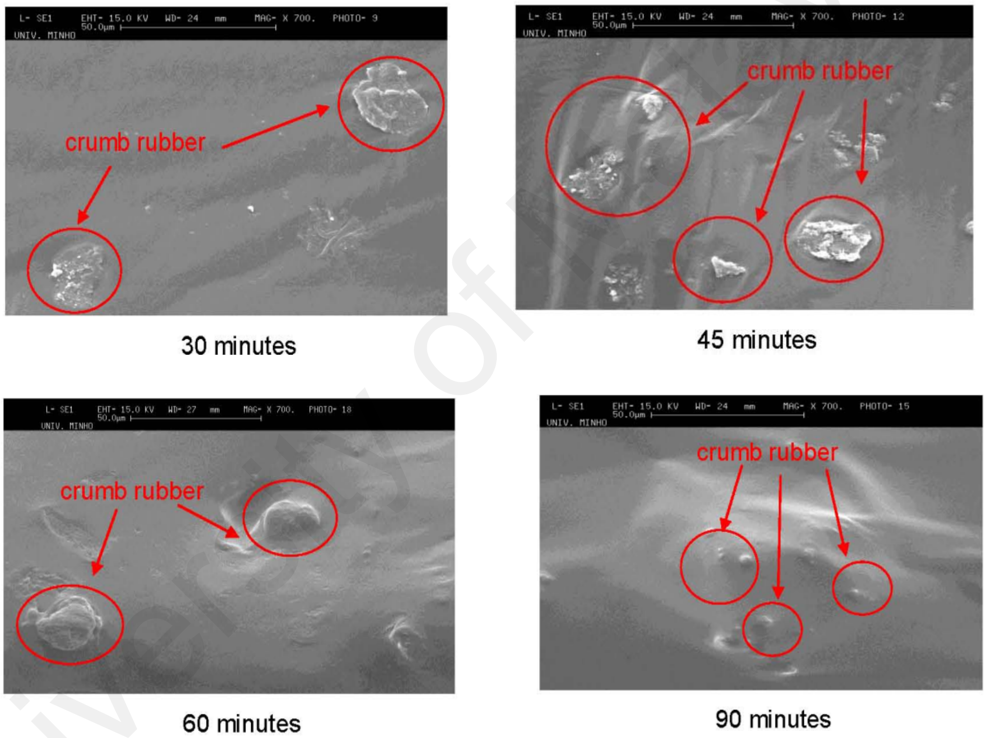
Another study showed that the time required to get the reaction of the rubber with the bitumen binder was dependent on various factors including the chemistry of the bitumen binder as well as the reaction temperature and rubber size. It showed that the blending time had no effect on rubberised bitumen binder properties (Putman *et al*., 2005).

A study by Shen and Amirkhanian (2005), on the effect of crumb rubber modifier (CRM) microstructures properties, showed that varying the mixing time from 15 to 45 min did not change the failure temperature, very much suggesting that a mixing time of 15 min was adequate to interact completely for 15% rubber with the binder.

According to a study by Paulo and Jorge (2008) who investigated the effect of blending conditions (time and temperature) on penetration, softening point and resilience modulus, stated that there was no significant effect using three blending time (45, 60 and 90 minutes) on modified binder properties although there was a tendency to become constant between 60 and 90 minutes.

Jeong *et al.* (2010) have reported from their study that the blending time and blending temperature for rubberised bitumen have a significant effect on the blending process which leads to enhanced binder properties. They also reported that longer blending time and high blending temperatures resulted in an increase in viscosity. This study has interesting results as it shows significant effects of both blending time and temperature on modified binder while showing an insignificant effect of same conditions on the control binder. Also, the reaction time is inversely proportional to the temperature of the material. Because of the high mixing temperatures, the bitumen binder during the process may results in primary aging, mainly due to oxidation of maltene compounds, and polymer degradation (Navarro *et al.*, 2009; Fawcett *et al.*, 2001; Airey, 2004).

Perez- Lepe *et al.* (2003) reported that mixing polymers into bitumen improved the mechanical properties of the modified binder, in tandem with the nature of polymer and the mixing processes employed.



30 minutes

45 minutes

60 minutes

90 minutes

Figure 2.4 Micrographs of the finished rubberised bitumen for different blending time (Paulo and Jorge, 2008).

Figure 2.4 shows the results of a study by Paulo and Jorge (2008) on the effect of blending time on rubberised bitumen process, from the micrographs of the finished rubberised bitumen binder. It is evident that relatively huge amounts of rubber are present in 30 and 45 minutes of reaction time. In 60 and 90 minutes of reaction time, the bitumen-rubber structures showed that the system is compatible. In 90 minutes of reaction time, the crumb rubber is fully incorporated in the bitumen binder.

Tortum *et al.* (2005) did a study on the determination of the optimum conditions of tyre rubber in asphalt concrete mixture with Marshal Test using the Taguchi method. This study investigated the effect of tyre rubber on asphalt pavement under different conditions/parameters of mixing time, mixing temperature, compacting temperature, rubber gradation, aggregate gradation and rubber content. The results showed that the most effective parameter on Marshal Stability is the rubber content, and the most effective parameter on flow is mixing time followed by mixing temperature. The study recommended that the optimum blending/ mixing time and temperature to be 5 minutes and 155 ^{o}C respectively.

2.12 Pavement Distress

Pavement distress is damage which appears in the hot mix asphalt (HMA) after construction due to repetition of traffic load, weather conditions, mix design, and lacke in quality control during construction. The road surface deficiencies of concern are:

- Permanent deformation (rutting)
- Fatigue cracking

2.12.1 Rutting Performance

Rutting is defined as longitudinal depression in wheel paths as a result of continued densification by traffic load (Figure 2.5). According to Sousa and Weissman (1994), rutting in bitumen pavement develops as load applications increases. Rutting seems as longitudinal depressions in the wheel paths with small upheavals on the sides. These are due to a combination of densification and shear deformation

Rutting is a primary measure of the performance of pavement in several pavement design methods. Rutting can occur as a result of problematic sub grade, unbound base course. Rutting failures are a consequence of heavy truckloads with high tyre pressures and high pavement temperatures. Hence, considerable selection of bitumen binder and aggregate combination will boost in providing optimum performing asphalt pavements (Sousa and Weissman, 1994).

Brown and Cross (1992) reported that permanent deformation in bituminous mixture is caused by consolidation and/ or lateral movement of the mixture under traffic. Shear failure (lateral movement) of the bituminous mixture courses generally occurs in the top 100 mm of the pavement surface. However, it can run deeper if proper materials are not used. Moreover, it was evident that rutting is caused mainly by deformation flow rather than volume change.

Figure 2.5 Permanent Deformations (Rutting)

Tayfur *et al.* (2007) claimed that after the initial densification, the permanent deformation of the bituminous mixture happens due to shear loads which take place occur close to the pavement surface which in fact is the contact area between the tyre and the pavement. These efforts increase without the volume variations in the bituminous mixture. They are the primary mechanisms in the development of rutting during the life span of the pavement design.

Increased permanent deformation or rutting has been related to the increase in truck tyre pressures, axle loads and volume of traffic (Brown and Cross, 1992). A study by Mahrez (1999) claimed that the use of rubberised bitumen binder has a significant effect on improving the mixture resistance to rutting deformation.

Permanent deformation of bituminous mixture is an intricate characteristic where the overall performance is controlled by aggregate (Sousa and Weissman, 1994). The properties of the binders that influence rutting are:

- Temperature susceptibility and rate of loading,
- Aging effects
- Moisture effects.

Rutting in flexible pavement can be divided into two types; consolidation rutting which happens with excessive consolidation of the pavement along the wheel path caused by decreased air voids in the asphalt concrete layer (Figure 2.6), or the permanent deformation of the base or subgrade. Instability rutting is due to the asphalt mixture properties and is an occurrence in the range of the top 2 inches of the asphalt concrete layer (Figure 2.7) (Sousa and Weissman, 1994).

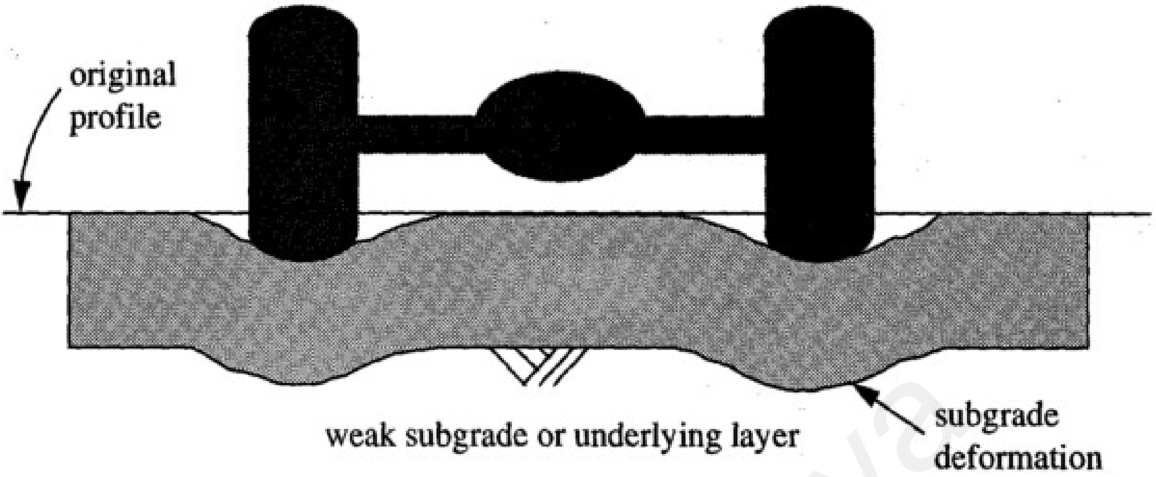


Figure 2.6 Consolidations Rutting of Flexible Pavement

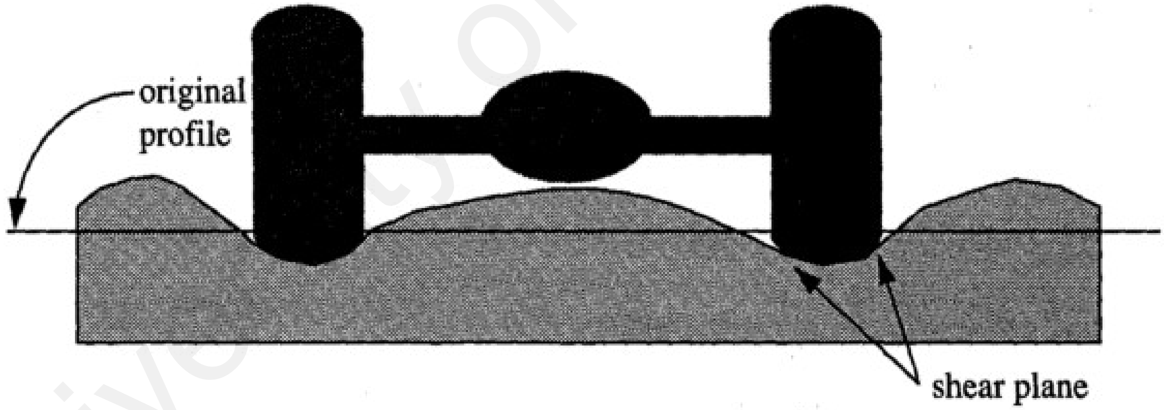


Figure 2.7 Instability Rutting of Flexible Pavement

2.12.2 Fatigue Cracking of Rubberised Bitumen

Fatigue is one of the most important distresses in asphalt pavement structure due to repeated load of heavy traffic services which occur at intermediate and low temperatures as shown in Figure 2.8.

A study by Aflaki and Memarzadeh (2011) investigated the effects of rheological properties of crumb rubber on fatigue cracking at low and intermediate temperature using different shear methods. The results showed that the high shear blending has more effect on improvement at low temperatures than the low shear blend.

The use of crumb rubber modified with bitumen binder seems to enhance the fatigue resistance, as illustrated in a number of studies (Raad and Saboundjian, 1998; Soleymani *et al*., 2004; McGennis, 1995; Biliter *et al*., 1997a; Hamed, 2010). The improved performance of bitumen rubber pavements compared with conventional bitumen pavements has partly resulted from improved rheological properties of the rubberised bitumen binder.

Figure 2.8 Fatigue Cracking

CHAPTER 3

MATERIALS AND RESEARCH METHODOLOGY

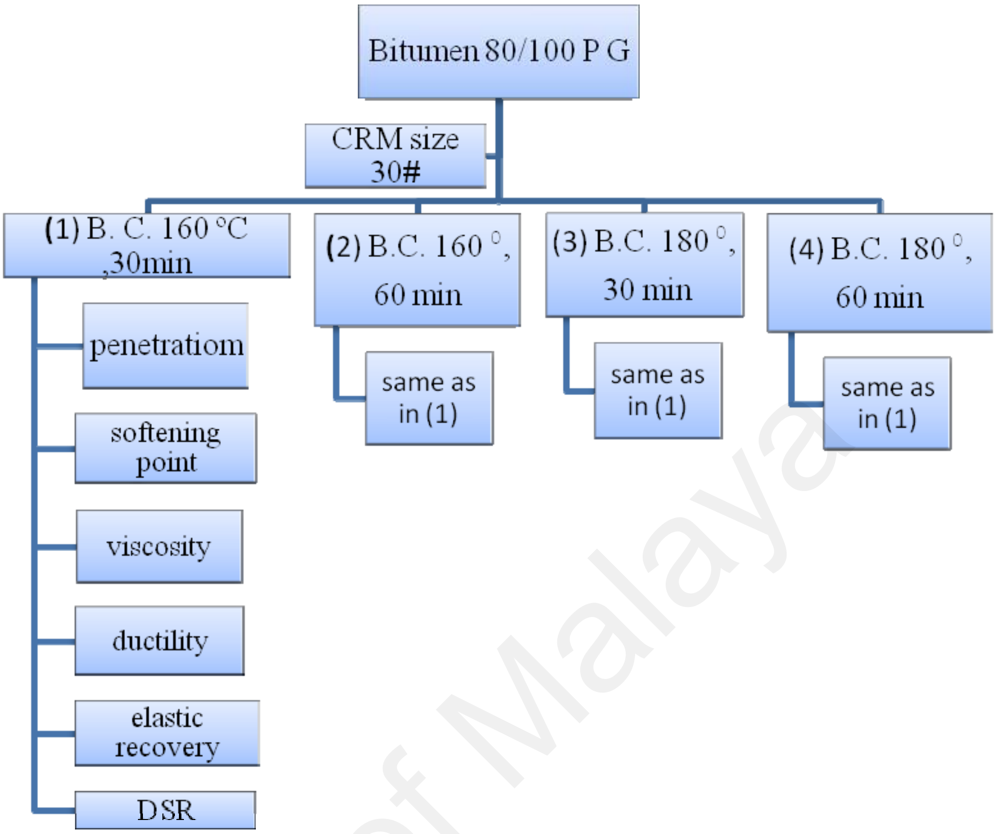
3.1 Introduction

The primary experimental aspect of this chapter focuses on the rheology and durability of rubberised bitumen binder. The factors that affect the rheology of bitumen binder were investigated by laboratory work under different blending conditions (blending temperatures and blending time) and different rubber content percentages by binder weight of modified and unmodified bitumen before and after aging. Several binder tests were conducted in achieving the objectives of the research study (see Figure 1.3).

Developing an effective rubberised bitumen binder is an experimentally iterative process. Assuming a predetermined choice of components as well as a large number of parameters (e.g., crumb rubber content, blending temperature, and blending time) are considerations accounted for in any investigation on the bitumen binder in order to select the optimum combination of parameters.

The durability of rubberised bitumen binder was investigated by binder tests after aging the binder. The effect of rubber content on the hardening of the rubberised bitumen binder was studied in order to produce pavement roads that could resist permanent deformation, induce less temperature susceptibility and offer long-life services, safety and cost effectiveness. All laboratory test methods and procedures were referred to ASTM standards and protocols (see the flow chart in parts A and B).

Part A:



Part B:

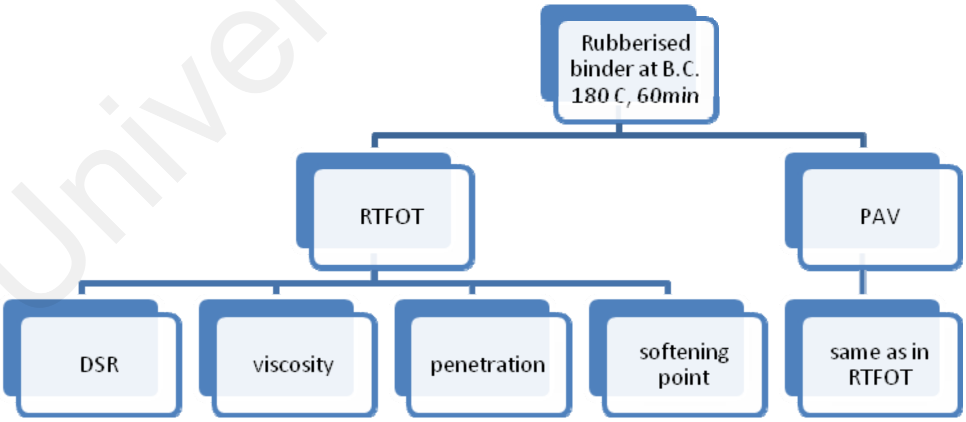


Figure 3.1 : Flow chart of Laboratory testing program

3.2 Rubberised Bitumen Materials

The rubberised bitumen binder was produced by mixing 80/100 penetration grade bitumen with different percentages of fine crumb rubbers. The rubber particle size passed 30 mesh sieve (0.6 mm) with specific gravity equal to 1.161.

3.2.1 Bitumen Binder

Bituminous binder of 80/100 penetration grade was used in this study. Table 3.1 shows the characteristics of the bitumen in this research.

Table 3.1: Properties of Base Binder Grade 80/100 Penetration

Test properties	Test result
Viscosity @135 ˚C (pas)	0.65
G*/ sin δ @ 64˚C (kpa)	1.35
Ductility @ 25 ˚C	100
Softening point @ 25 ˚C	47
Penetration @ 25 ˚C	88

3.2.2 Crumb Rubber Modifier (CRM)

For the purpose of maintaining consistency of the CRM throughout the entire study, one batch of crumb rubber obtained from one source was solely used as displayed in Figure 3.1. In this study, fine crumb rubber size 30 # (0.6 mm) was selected in order to reduce segregation (Liu *et al*., 2009; Shen *et al*., 2004). CRM gradation and chemical components are illustrated in Tables 3.2 and 3.3 respectively.

Table 3.2 : Sieve Analysis of Tyre Rubber

Sieve no. (size, mm)	Particle size distribution % passing
No. 10(2.00)	100
No. 16(1.180)	100
No. 20(0.850)	100
No.30 (0.600)	97.7
No.40(0.425)	61.1
No. 50(0.300)	33.9
No. 80(0.180)	12.5
No. 100 (0.150)	7.5
No. 200(0.075)	0.0

Table 3.3: Chemical Components of Tyre Rubber

Major rubber components	Tire rubber
Acetone extract (%)	11.0
Rubber hydrocarbon (%)	50.5
Carbon black content (%)	32.5
Natural rubber content (%)	34.0
Ash content (%)	6.0

Figure 3.2: Crumb Rubber size (0.6 mm)

3. 3 Mixing Preparation and Procedures

A propeller mixer was used to prepare the rubberised bitumen as shown in Figure 3.2 and the wet process was utilised. Binder mixing was conducted at the velocity speed of 200 rpm. Two different blending temperatures (160 °C and 180 °C) and two different blending times (30 and 60 minutes) were also used in tandem with the blending time as recommended by Jeong *et al.*(2010). Five different percentages of crumb rubber by binder weight (0%, 4%, 8%, 12%, 16% and 20%) were selected. The steps involved in the preparation of samples are as follows:

- The bitumen was heated in the oven until it was converted into liquid. The temperature was approximately 100 °C.
- The molten bitumen was poured into a 1L cylindrical tin (i.e., two-thirds of its volume). Prior to this, an empty tin was weighed using an electronic weighting balance. After pouring the molten bitumen, the cylindrical tin with the bitumen was weighed again to obtain the nett weight of the bitumen.
- The required amount of crumb rubber used, as a function of percentage for mass of the bitumen was obtained.
- Depending on the required sample, the bitumen was heated to 160 °C or 180 °C. Then, the hot plate was heated to the desired temperature prior to using the propeller mixer.
- Once the oven temperature reached 160 °C or 180 °C (i.e., by using a stainless steel thermometer), the bitumen sample was taken out from the oven. The crumb rubber percentage amount was added immediately to the hot bitumen.
- A mechanical stirrer was placed centrally in the molten rubberised bitumen at the speed rate of 200 rpm at the duration blending time of 30 or 60 minutes.

- Finally, the rubberised bitumen binder sample was obtained. It is important to note that the samples were covered and stored safely prior to testing.

Figure 3.3: Propeller mixer used during the rubberised binder preparation

3.4 Bitumen Binder Testing

The binder tests used in this study include:

- Standard Test Method for Softening Point of bituminous materials (ASTM D36).
- Standard test method for Penetration of bituminous materials (ASTM D5).
- Standard test method for Viscosity determination using the Brookfield Thermosel Apparatus (ASTM D4402).
- Proposal test method for determining the rheological properties of bituminous binder for specification purposes using a Dynamic Shear Rheometer (DSR) (ASTMD-4 proposal P246).

- Standard test method for Ductility of bituminous materials (ASTM D113).
- Standard test method for Elastic Recovery of bituminous materials by ductilometer (ASTM D6084).
- Standard test method for the effect of heat and air on a moving film of asphalt (Rolling Thin – Film Oven Test) (ASTM D 2872).
- Practice for accelerated ageing of bituminous binder using a Presser Ageing Vessel (PAV) (ASTM D 6521).

3.5 Softening Point Test (Ring and Ball) (ASTM D 36)

According to the specification test, softening point is the temperature at which the bitumen reaches a particular degree of softening. This is tested using a ring and ball apparatus as shown in Figure 3.3. A brass ring containing test sample of bitumen is suspended in liquid such as water or glycerine at a specific temperature. A steel ball is put on the bitumen sample and the liquid medium is heated at 5˚C per minute. Temperature is recorded when the softened bitumen touches the metal plate which is at a specified distance of 25 mm. Often, higher softening point indicates lower temperature susceptibility and is found in hot climates. This test method focuses on the determination of the softening point of bitumen ranging from 30˚C and 157˚C.

3.5.1 Signification and Use

According to ASTM D36 (2006), bitumen is a visco-elastic materials without specifically defined melting points. They slowly become softer and less viscous as temperature is elevated. Thus, softening point has to be determined by an arbitrary and closely defined method for results to be reproduced.

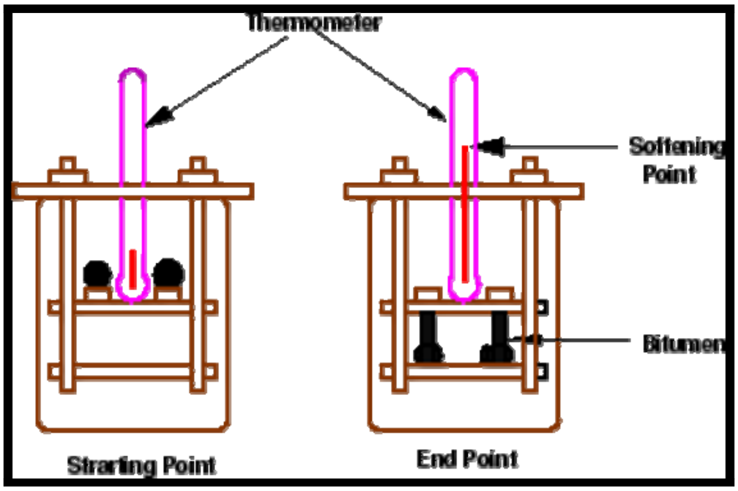


Figure 3.4: Softening Point Test Setup

3.5.2 Specimen Preparation

The bitumen was heated until it becomes sufficiently fluid to pour. The bitumen sample was stirred carefully in order to avoid the incorporation of air bubbles. Two brass rings were heated and then placed on the metal plate furnished with release agent. The bitumen was then poured into each ring, and then the specimen was allowed to cool at room temperature for about 30 minutes, and then the surplus bitumen was then cut off, levelled with the top of the rings by using a heated spatula.

Figure 3.5: Softening Point Test Apparatus

3.5.3 Test Procedure

The apparatus which were assembled in the laboratory: hood with the specimen rings, ball centring guides and thermometer placed in a 600 ml beaker filled with 105 mm distilled water (see Figure 3.4). Ice was used to maintain the water temperature at 5 ˚C at the beginning of the test. During the test, the water temperature was maintained at 5 ˚C for 15 minutes. A steel ball was placed on each of the test specimen. Later, the beaker was heated using the gas burner meanwhile the water was stirred from time to time to keep the temperature at a uniformed rate of 5 ˚C per minute. The heating of the bitumen samples was continued until it softened and for the ball to pass through the ring. For each ring and ball, the temperature was measured with the thermometer the instant the bitumen surrounding the ball touched the bottom plate. This was taken as the softening point of bitumen.

3.6 Penetration Test (ASTM D5)

The purpose of this test is to examine the consistency of bituminous materials by measuring the distance (tenths of a millimetre) when a standard needle is vertically penetrated into the bitumen sample under standard specification conditions of loading, time and temperature. Higher penetration values indicate softer consistency. The penetration test is a common test for the purpose of defining the various grades of bitumen. Form past research, it is evident that reduction in penetration value indicates resistance of binder to permanent deformation such as rutting.

3.6.1 Scope

The test method covers determination of the penetration of solid and semi solid bituminous materials. Penetration presents the consistency of bituminous material expressed as the distance in tenth of a millimetre when a standard needle vertically penetrates a sample or the materials under known conditions (5 sec, 25 ˚C and 100g).

3.6.2 Apparatus

As shown in Figure 3.5 the penetration apparatus consists of a needle, needle holder, sample container, water bath, transfer dish, timing device and thermometer.

Figure 3.6: Penetrometer Apparatus

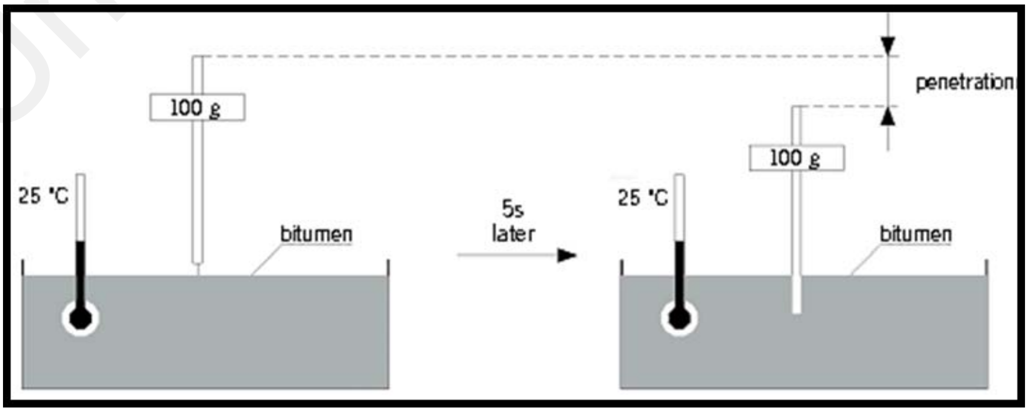


Figure 3.7 : Penetration Test Setup (Branco *et al*., 2005)

3.6.3 Test Procedure

The bitumen was heated in the oven until it becomes sufficiently fluid, thoroughly poured into a slandered penetration tin and allowed to cool for 60 minutes. Then, the containers were put in a water bath for about 1.5 hour. The needle holder and the guide were examined to establish the absence of water and other extraneous matter. The needle with toluene, dried with a clean cloth was inserted into the penetrometer.

The sample container was then placed in the transfer dish. The container was covered completely with water bath at a constant temperature of 25 ˚C and then, the transfer dish was placed on the stand of the penetrometer. The needle was positioned by slowly lowering it until its tip just made contact with the surface of the sample. This was accomplished by bringing the actual needle tip into contact with its image reflected on the surface of the sample from a property placed source of light. Then, the pointer was brought to zero point and quickly released for the specified period of 5 seconds as shown in Figure 3.6. The reading of the dial was recorded. The distance penetrated by the sample in tenth of milimeter was computed by subtracting the final reading of the dial with the zero point.

3.7 Viscosity Determination of Unfilled Asphalt Using The Brookfield Thermosel Apparatus (ASTM D 4402- 87)

3.7.1 Scope

This test method uses the Brookfield Thermosel apparatus for measuring the apparent viscosity of bitumen from 38 to 260 ˚C (100 to 500 ˚F).

3.7.2 Significance and Use

The Brookfield Thermosel Viscometer can be used to measure the apparent viscosity of bitumen material at application temperatures. Some bitumen may exhibit non- Newtonian behaviour under the conditions of this test or at temperature within the range of this method. Since non- Newtonian viscosity values are not unique material properties but reflect the behaviour of the fluid and the measurement system, it should be recognised that measurements made by this method may not always predict performance under the condition of use. Comparison between non Newtonian viscosity values should be made only for measurements made with similar viscometer under similar conditions of shearing stress and shear history.

3.7.3 Apparatus

Brookfield Thermosel high temperature viscosity measuring system is shown in Figure 3.7, which includes spindles, thermoses system; thermo container and sample chamber, controller and graph plotting equipment.

Figure 3.8: Brookfield Viscometer Apparatus

3.7.4 Sample Preparation

For not more than 30 min, the sample was heated in the oven. The sample was then poured into the sample holder to approximately 10 ± 0.5 g. The sample was cooled in room temperature before transferring to Brookfield Thermosel for testing.

3.7.5 Test Procedure

The Thermsel power was turned on. The proportional temperature controller was set to desired test temperature (90 ˚C). Using the extracting tool, the sample holder was then placed into the thermo container. The viscometer was lowered and the thermo container aligned. Spindle No. 27 (used in this research study) was inserted into the liquid in the chamber and was coupled with the viscometer. Then, the sample was allowed to wait for 15 minutes until it reached the equilibrium temperature of 90 ˚C. The spindle was rotated at a lower speed (10 – 15 rpm) and then increased to 20 rpm when the torque starts dropping. The reading for viscosity was taken when the torque reading was stable. The average of the three readings were taken at 60 seconds apart at each test temperature was recorded. The test procedure as mentioned above, were followed for each test temperature. The test temperatures used in this research were 90, 100, 130, 135, 150 and 170 ˚C.

3.8 Test Method for Determining the Rheological Properties of Bituminous Binder for Specification purposes using a Dynamic Shear Rheometer (DSR) (ASTM D-4 proposal P246)

3.8.1 Scope

This proposal test method focuses on determining the linear visco-elastic properties of bitumen when tested in dynamic (oscillatory) shear using parallel plate test geometry as shown in Figures 3.8 and 3.9.

3.8.2 Test Method

This proposed standard contains the procedure used to measure the complex shear modulus (G*), storage modules (G´), or the loss modules (G´´), phase angle (δ) and rutting factor, G*/sin δ of bitumen binders using a dynamic shear rheometer and parallel plate test geometry. Test specimens of 1 mm thick by 25 mm in diameter were formed between parallel metal plates. During testing, one of the parallel plates was oscillated with respect to the other at pre-selected frequencies and rotational deformation amplitudes. The below is formula to calculate the G*, G', G'' as well as δ in Eqn. 3.1, 3.2, 3.3 and , respectively :

$$G^* = (\tau / \gamma) \qquad \text{Eqn. (3.1)}$$

$$G' = \cos(\delta)\,(\tau / \gamma) \qquad \text{Eqn. (3.2)}$$

$$G'' = \sin(\delta)\,(\tau / \gamma) \qquad \text{Eqn.(3.3)}$$

$$\delta = G' / G'' \qquad \text{Eqn.(3.4)}$$

where:

G* : complex shear modulus

τ : shear stress

γ: shear strain

G': storage modulus

G'' : loss modulus

δ: phase angle.

Rutting factor $G^*/\sin \delta$ (superpave parameter) was set as the stiffness indicator for measuring and evaluating the rutting resistance of both unmodified and polymer-modified binders (Bahia and Anderson, 1995a). SHRP has defined the requirements of rutting factor $G^*/\sin \delta$, which correspond to the high-temperature viscous component of binder stiffness.

The required amplitude depends upon the value of the complex shear modulus of the bitumen binder being tested. The required amplitudes were selected to ensure that the measurements were within the region of linear behaviour. The test specimen was maintained at the test temperature to within ± 0.1 ˚C by positive heating and cooling of the upper and lower plates. Oscillatory loading frequencies using this proposed standard range from 1 to 100 rad/s using a sinusoidal waveform. Specification testing was performed at a test frequency of 10 rad/s. The complex modulus (G^*) and phase angle (δ) are calculated automatically as part of the operation of the rheometer using proprietary computer software supplied by the equipment manufacturer.

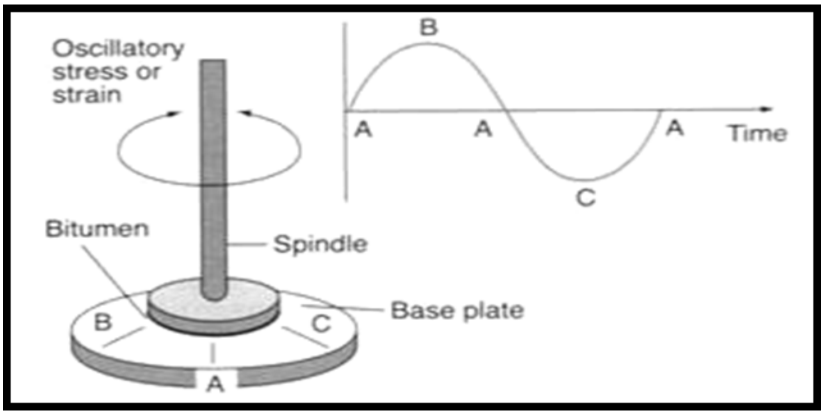


Figure 3.9: Schematic diagram of DSR mode test.

3.8.3 Significance and Use

The test temperature is linked to the temperature experienced by the pavement in the geographical area for which the bitumen binder is intended. The complex shear modulus is an indicator of the stiffness or resistance of bitumen binder to deformation under load. The complex modulus and phase angle define the resistance to shear deformation of the bitumen binder in the linear visco-elastic region. Other linear visco-elastic properties, such as the storage modulus (G´), or the loss modulus (G´´), can be calculated from the complex modules and the phase angle. The loss modulus (G´´) is a measure of the energy dissipated during each loading cycle.

3.8.4 Preparation of Apparatus

The apparatus were prepared in concurrence with the manufacturer’s recommendation. The test plates were mounted on the test fixtures and tightened firmly. The test plate’s surface was wiped clean and dried before mounting the plates; this was to ensure that the specimen will adhere to both plates uniformly and strongly.

Figure 3.10: DSR Apparatus

3.8.5 Preparation of Samples and Test Specimens

The bitumen binder was heated until it was adequately fluid to be poured in the required specimens and allowed to anneal. Prior to testing, the annealing molecular associations (hardening) that occurs during normal storage at ambient temperature were removed. The sample was covered and stirred occasionally during the heating process to ensure homogeneity and to remove air bubbles. The heating temperature and time were minimised to avoid hardening the sample.

The sample was conducted using a temperature sweep. Thus, in this study work the range was from 30 ˚C to 80 ˚C. The temperature of the liquid bath was about 30 ˚C. The removable upper plate was tight on the DSR machine. The zero point would be indicated automatically by the computer software. The upper plate was imposed into the sample to obtain a thickness of 1mm; the sample covered the whole surface of the plate. The upper plate was reassembled into the rheometer, using the software, the plates were then moved together to squeeze the specimen mass which was between the two plates. When measurement position was achieved, the valve was turned on; allowing the liquid bath to fill in the environment chamber and then the testing was started.

3.9 Ductility Test (STM D113- 99)

3.9.1 Scope

The ductility of a bituminous material is measured by the distance of its elongation before breaking when two ends of a briquette specimen of the material are pulled apart at specified speed and temperature. The specified conditions of the tests are temperature of about 25 ˚C ± 0.5 ˚C and the speed of 5 cm/min.

3.9.2 Significance and Use

This test method provides one measure of tensile properties of bituminous materials and can also be employed to measure ductility for specification requirements. Ductility is considered as an important elasticity property of asphalt mix, thus would indicate the adhesiveness of bitumen binder mix, like the capability of the bitumen binder to resist deformation under high temperature load services. Factors which greatly affect the ductility value include pouring temperature, test temperature, rate of pulling etc.

3.9.3 Preparation of Specimen

The mould was assembled on a brass plate. The mould design is similar to that described in the standard test method for ductility test (ASTM D113). Figure 3.10 shows the ductility samples. The surface of the plate and the interior sides of the mould were coated with a layer of glycerine and talc to avoid sticking of the materials. The bituminous sample was heated until it becomes sufficiently fluid. Then, the bitumen was poured into the ductility mould with continuous stirring and high care to avoid disarranging the parts and thus distorting the briquette.

The mould containing the test specimen was allowed to cool at room temperature for a duration time of 40 minutes and then it was placed in the water bath at the specified temperature of 25 ˚C for 30minutes. The volume of water in the ductility machine was about 10 L, and the specimen was immersed to a depth of 10 cm and/supported on a perforated shelf by 5 cm from the bottom of the bath. Finally, the excess bitumen was cut and removed by a hot straight edged putty spatula to make the mould full in size.

3.9.4 Test Procedure

The brass plate and mould with briquette specimen was placed in the water bath and kept at the specified temperature of 25 ˚C for a period of 85 to 95 min prior to testing. Then, the briquette was removed from the plate, the side pieces were detached. The rings were attached at each end of the clips to the pins or hook in the testing machine and the two clips were pulled apart at uniform speed as specified until the briquette ruptured. The distance in centimetres through which the clips have been pulled to produce rupture was measured as ductility as presented in Figure 3.11.

Figure 3.11: Ductility Briquette Samples

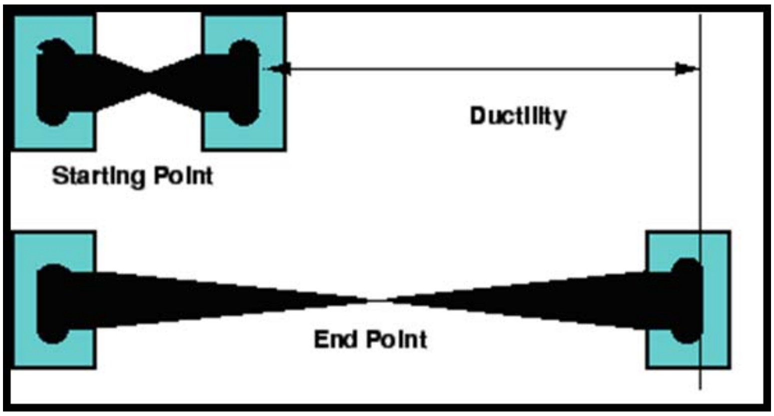


Figure 3.12: Ductility Test Measurement

3.10 Elastic Recovery Test (ASTM D 6084- 97)

3.10.1 Scope

This test method presents the elastic recovery of a bituminous material measured by the recoverable strain determined after severing an elongated briquette specimen of the material. These specimens are pulled to a specified distance at a specified speed and temperature. The elongated distance considered is 10 cm; the test was conducted at temperature of 25 ˚C ± 0.5 ˚C and with a speed of 5 cm/min ± 5 %.

3.10.2 Significance and Use

This test method is advantageous in verifying that the added material to the bitumen provides a significant elastomeric characteristic.

3.10.3 Procedure of the Test

This test is the same as the ductility test whereby the same test apparatus is used with the exception for the mould design which is similar to the description in the elastic recovery standard test (ASTM D6084).

The mould was assembled on the brass plate. The surface of the plate and the interior surface of the sides of the mould are thoroughly coated with a thin layer of a mixture of glycerine, dextrin and talc to prevent the test materials from sticking. The specimen and the mould were placed in the water bath at the specified test temperature of 25 ˚C for 90 ± 5 min prior to testing. The specimen was removed from the plate by shearing action between specimen and plate, avoiding any bending of the test specimen. The side pieces were removed carefully in order not to distort or fracture the specimen. The specimen was attached to the testing machine. The ring at each end of the clips was attached to the pins or hooks in the testing machine. The two clips were pulled apart at a uniform speed of 5 cm/min to an elongation of 10 ± 0.25 cm. Once the elongation stopped then, the test specimen was cut into two halves at the midpoint using a scissors. The specimen was allowed to remain in the testing machine in an undisturbed condition at the specified temperature for a period of 60 min. After 60 minutes, the travelling carriage was moved back carefully to a position where the ends of the specimens just touched. The total length of the specimen with the severed ends just touching each other was recorded.

3.10.4 Calculation and Report

The percent recovery is calculated as follows:

$$\text{Recovery, \%} = (E - X) / E \times 100 \qquad \text{Eq. (3.5)}$$

Where:

E = original elongation of the specimen (10 cm)

X = elongation of the specimen with severed ends just touching, cm

3.11 Ageing of The Binder Using The Rolling Thin Film Oven Test (RTFOT) (ASTM D 2872-88)

3.11.1 Test Equipment

The RTFOT procedure requires an eclectically heated convection oven to be heated to the ageing temperature of 163 ˚C. The oven is inside a vertical circular carriage which can hold up 8 horizontally positioned, cylindrical bottles which may be rotated mechanically around the carriage centre. An air jet blows into each bottle whenever it passes through its position on this carriage during the circulation.

3.11.2 Test Procedure

A bituminous binder sample was heated until it becomes fluid. The RTFOT bottles were filled with 35 ± 0.5 gm of binder. The RTFOT oven (Figures 3.12 and 3.13) was prepared at the ageing temperature of 163 ˚C ± 0.5 ˚C, for a period of 16 hours prior to use. The thermostat was set so the oven will return to this temperature within 10 minutes after the sample bottles were loaded.

The test bottles were loaded into the carriage, with any unused slots filled with empty bottles. The carriage was started and rotated at a rate of 15 ± 0.2 rev/ min. The air flow was set at a rate of 4000 ± 200 ml/min. The samples were maintained under these conditions for 85 minutes.

When the test bottles (Figure 3.14) were rotated, the melted bitumen will form a thin film layer inside the bottle thus providing a defined wide surface area, out of any volatile reaction thus allowing it to easily evaporate and also oxygen can react with the bitumen layer. The bitumen was aged for a period of 85 min with an air flow rate of

4l/min. The RTFOT greatly accelerates and standardises the ageing process of bitumen and leads to specimens with a defined protest history. The bitumen from the bottles was poured out to prepare specimens for binder tests.

Figure 3.13: Rolling Thin Film Oven (RTFO)

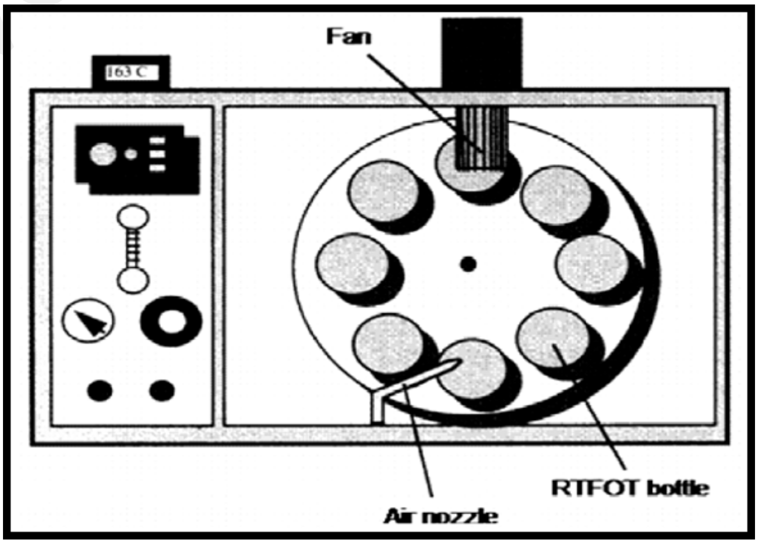


Figure 3.14: RTFOT schematic drawing

Figure 3.15: RTFOT Bottles

3.12 Pressure Ageing Vessel Test (PAV) (ASTM D 6521)

The pressure ageing apparatus consists of the ageing vessel and a temperature chamber. Air pressure is provided by a cylinder of dry, clean compressed air with a pressure regulator, release valve and a slow release bleed valve. The pressure ageing vessel exposes the asphalt to a simultaneous high pressure of 2.1 MPa and high temperatures above 100 ˚C for a period of 20 hours. The vessel must accommodate at least 10 sample pans by means of a sample rack. Figure 3.15 presents the Pressure Ageing Vessel Oven (PAV).

3.12.1 Preparation of The Specimen

In the PAV test, bitumen specimen of 50gm which had been previously aged in the RTFO, was melted and poured into sample pans.

3.12.2 Test Procedure

First, the temperature chamber (oven) was turned on. The vessel was placed in the chamber and allowed to set at 100 ᴼC temperature. The pressure air vessel pans are placed in the sample rack. When the test temperature has been achieved, the vessel was removed from the oven and the samples rack was placed in the hot vessel. The lid was installed and the shear ring assembly was secured. This step should be completed quickly to avoid excessive loss of vessel heat. The vessel was loaded into the temperature chamber and the pressure hose and temperature transducer were coupled with their respective mates. When the vessel temperature was within 2 ˚C of the test temperature, air pressure was applied using the valve on the air cylinder regulator. When timing of the test begins, the air pressure was applied.

After 20 hours, the pressure was slowly released by the bleed valve. In general, 8 to 10 minutes will be required for pressure release. If the pressure was released faster, excessive air bubbles will be present in the sample. The pans were removed from the samples holder and placed in a different oven for 30 minutes at 163 ˚C, in order to get rid of the entrapped air from the samples. The samples which are aged were then transferred to a container that stores the material for testing.

Figure 3.16: Pressure Ageing Vessel Oven (PAV)

3.13 Analysis of Variance (ANOVA) Test

Analysis of Variances (ANOVA) is a statistical method that is used for assessing relationships and differences among the means of two or several data sets. It is a guide for determining whether or not differences in a set of counts or measurements were most likely due to the modern chance variation. In this study, ANOVA was performed using Two Factor without replication: this performs an analysis of variance between two or more data sets. Normally, it is used when there is only one sample from each data set.

The significant level of the data sets is determined by evaluating the F- ratio and comparing it to the F- critical value for the samples. If the F- ratio (F- Statistic) is larger than F- critical, then the variation between the groups is statistically significant. The observed P- value is the probability of observing the F- ratio or larger when the mean test results are equal. If the P- value is less than the desired level of significance (α), then the corresponding variant becomes significant. The level of significance (α) used in this research was 0.05 that represented a probability of 5% of the hypothesis represented, thus, the model may not be true (Mahrez, 2008)

CHAPTER 4

RESULTS ANALYSIS AND DISCUSSION

4.1 Introduction

This chapter aims to compare the performance of the rubberised bitumen binders prepared under different blending temperatures and blending times, as well as various percentages of rubber crumb content. This chapter also presents the performance of rubberised bitumen after aging using different mixing methods and different rubber crumb contents in order to investigate the physical and rheological properties of rubberised bitumen.

4.2 Analysis and Discussion of Penetration Results

The penetration results of different blending temperatures and blending times for the various crumb rubber contents are shown in Table 4.1 and illustrated in Figures 4.1.–4.4.

Table: 4.1 Penetration Results of Rubberised Bitumen Specimen of Different Blending Conditions

Penetration value (0.1 mm)							
Time of mixing	CRM	160 °C			180 °C		
		Mean	Std. Dev.	n	Mean	Std. Dev.	n
30	0	89.67	2.08	3	89.33	1.53	3
	4	79.67	2.52	3	76.00	1.00	3
	8	67.67	2.52	3	63.67	3.06	3
	12	54.67	2.52	3	50.00	2.00	3
	16	49.00	6.56	3	41.67	3.06	3
	20	37.67	2.52	3	34.33	3.06	3
60	0	88.00	2.00	3	88.67	3.06	3
	4	77.67	2.52	3	70.67	2.52	3
	8	65.33	4.04	3	60.67	2.52	3
	12	52.67	2.52	3	47.67	2.52	3
	16	43.67	2.52	3	40.67	2.52	3
	20	33.33	4.16	3	32.00	3.00	3

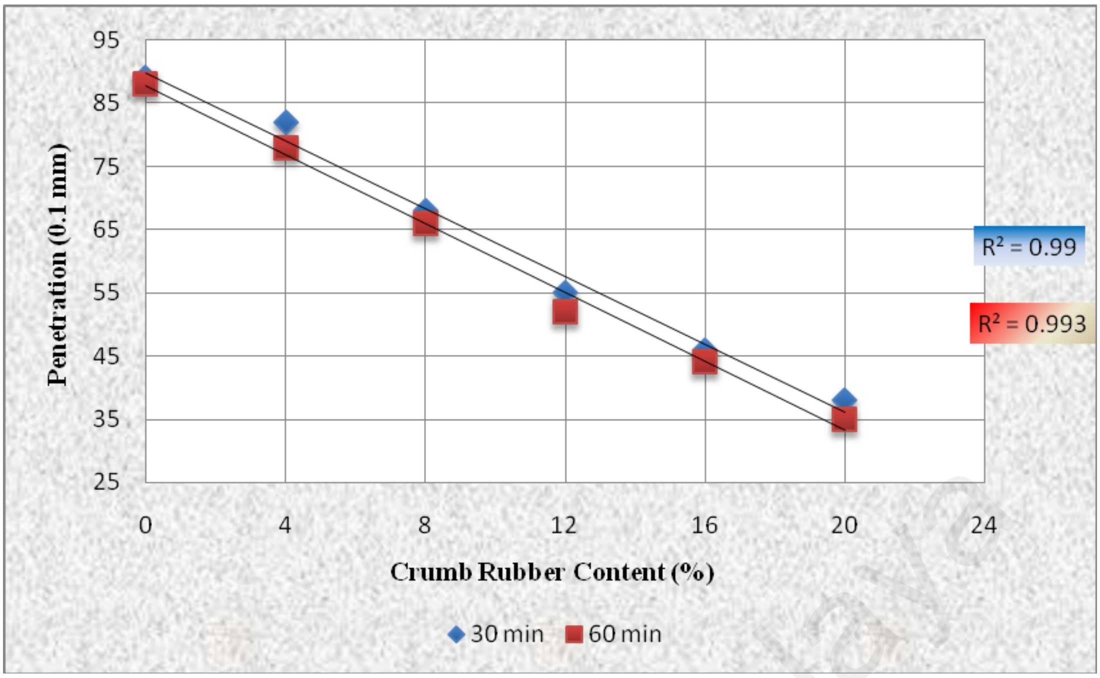


Figure 4.1: Penetration Results vs. Blending Time for Specimen with Blending Temperature of 160˚C

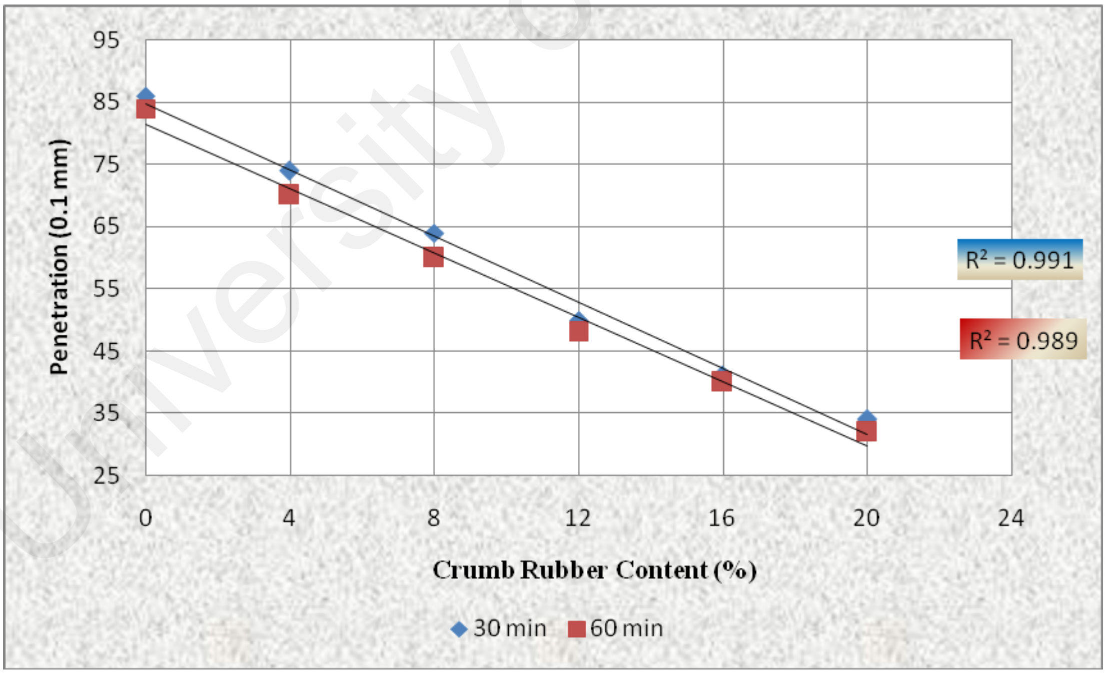


Figure 4.2: Penetration Results vs. Blending Time for Specimen with Blending Temperature of 180 ˚C

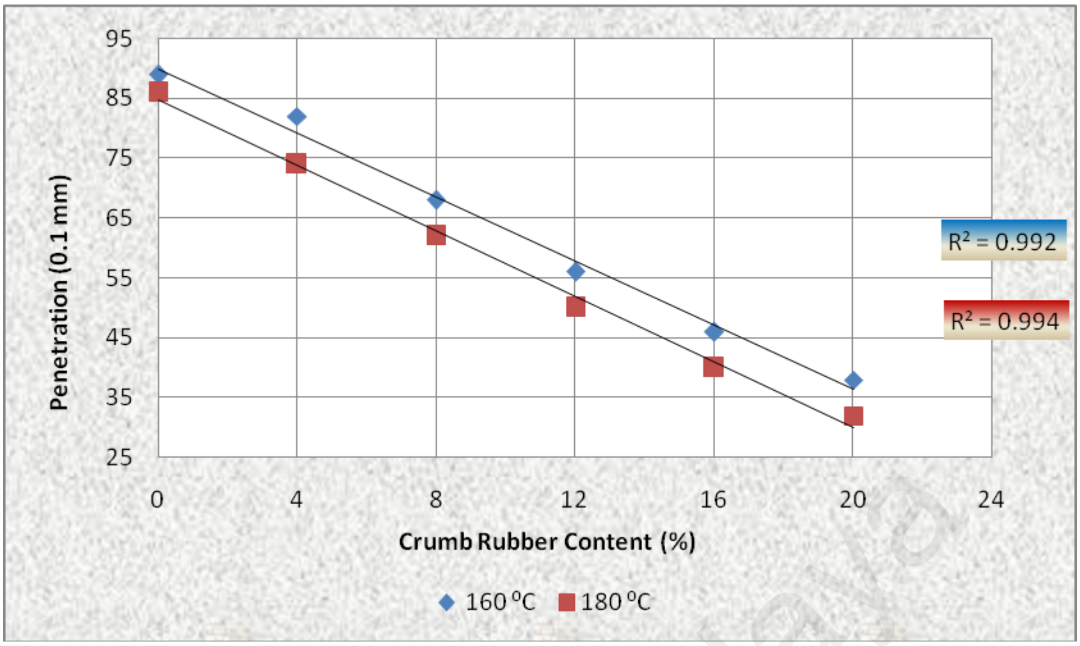


Figure 4.3 : Penetration Results vs. Blending Temperature for Specimen with Blending Time of 30min

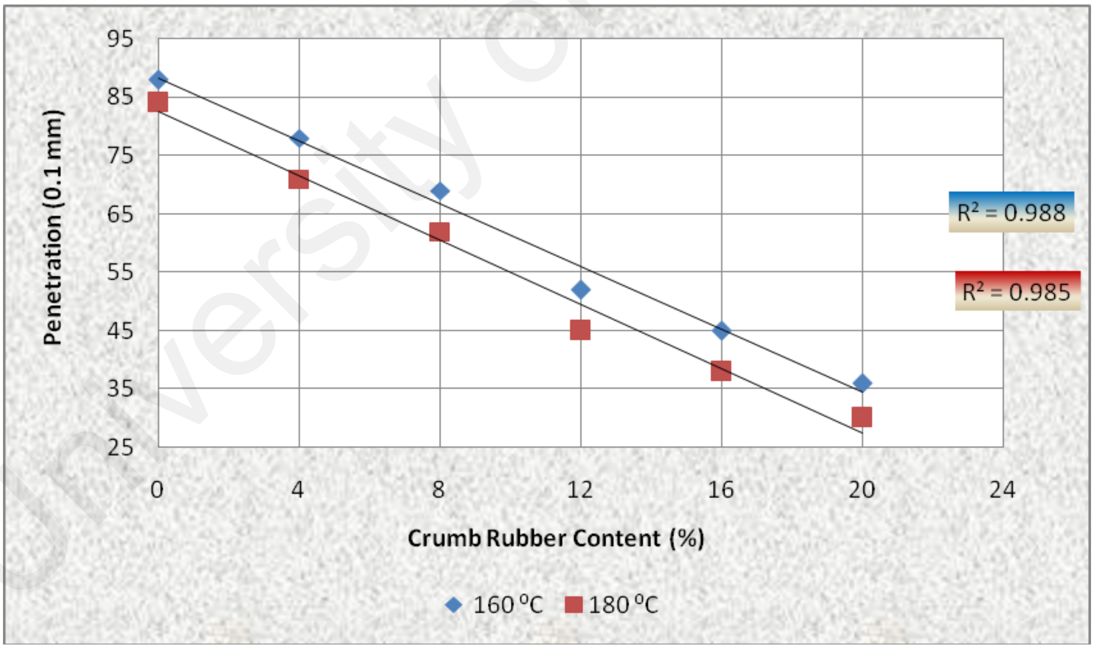


Figure 4.4 : Penetration Results vs. Blending Temperature for Specimen with Blending Time of 60min

4.2.1 Effect of Crumb Rubber on Penetration

Figures 4.1- 4.4 show a dramatic decrease in penetration as the rubber content increased in the bituminous specimens. The decrease is linearly dependent with strong correlation coefficient factor about 0.993- 0.989 for all the figures. The results showed that the higher crumb rubber content in the mix led to lower penetration values, however , the results of this research agree the finding of previous studies (Katman, 2006; Kumar *et al*. ,2009). These results are due to the crumb rubber content exhibiting a strong effect on penetration reduction by increasing the stiffness of crumb rubber modified bitumen binder. This would make the binder more resistant to high temperature susceptibility, thus leading to high resistance to permanent deformation such as rutting as mentioned by Liu *et al*. (2009) and Huang (2008).

From Figures 4.1- 4.4, it is evident that the averages decrees in penetration of modified binder samples compared with unmodified binder were approximately about 17 to 67% for 4 and 20% rubber content respectively.

Statistical analysis of ANOVA indicates a significant difference of crumb rubber content on penetration test for different blending conditions with (p–value) less than the assumed value of 0.05 and the F ratio was more than F critical as displayed in Tables 4.1 -4.4.

4.2.2 Effect of Blending Time on Penetration

The variation in penetration values due to varied blending times did not show any significant difference in penetration values for all samples (unmodified & modified), for mixing temperatures of 160 °C and 180 °C, respectively as shown in Figures 4.1 and 4.2. The findings showed that there were no significant differences in the penetration results at blending times 30 min and 60 min respectively, when tested at different temperatures (160 ºC and 180 ºC).

This finding is consistent with the findings of Moreno *et al*. (2011) who presented that the blending time has an insignificant effect on the physical properties of the modified bitumen binder. Also according to Paulo and Jorge (2008), the results showed insignificant effect of blending time of crumb rubber modified bitumen on penetration test for varying blending time. Moreover, the time required to get the reaction of the bitumen with the rubber was dependent on several factors including the chemistry of the bitumen binder as well as blending temperature and crumb rubber size (Putman *et al*., 2005). Thus, from all these findings, engineers need not be concerned about the blending time between 30 – 60 minutes as it does not have any major effect on the properties of modified binder.

From statistical analysis of ANOVA, it is evident that blending time has insignificant effect on penetration test for modified and unmodified bitumen samples as illustrated in Tables 4.1 and 4.2, respectively.

4.2.3 Effect of Blending Temperature on Penetration

The variation in penetration due to varied blending temperature shows significant effect for all modified samples, although, it displayed insignificant influence on unmodified bitumen for different mixing times of 30 min and 60 min, respectively as shown in Figures 4..3 and 4.4.

From statistic analysis ANOVA, it is evident that blending temperature has significant effect on penetration test for modified and unmodified bitumen samples as illustrated in Tables 4.3 and 4.4 respectively; it illustrates that the value of F ratio is higher in comparison to the value F critical and also, the P – value is smaller than (α = 0.05).

Figure 4.3 shows that the averages decrease in penetration of modified binder samples compared with unmodified binder were about 9 to 57 % for crumb rubber content of 4 and 20% respectively, at blending temperature of 160 °C, and were about 14 to 63 % for crumb rubber content of 4 and 20% respectively, at blending temperature of 180 °C. A similar trend was obtained in Figure 4.4 at blending time of 60 minutes. The average decrease in penetration of modified binder samples compared with unmodified binder were about 11 to 59 % for crumb rubber content of 4 and 20% respectively, at blending temperature of 160 °C, and were about 15 to 64 % for crumb rubber content of 4 and 20% at blending temperature of 180 °C.

The increase in blending temperature enhanced the particle size of the rubber and led to the increase in rubber mass through the interaction and swelling of the rubber into the bitumen during the blending process, which led to the decrease in the penetration values of rubberised bitumen samples (Lee *et al*., 2008; Jeong *et al*., 2010).

Table 4.2 Effect of CRM Binders and Blending Time at 160 °C on Penetration Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	4636.622	15	309.1081	4.695824	0.001125	2.014804
Blending Time	33333.5	2	16666.75	2.274543	0.3332	3.31583
Error	2509.114	30	83.63712			
Total	40479.23	47				

Table 4.3 Effect of CRM Binders and Blending Time at 180 °C on Penetration Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	4207.714	15	300.551	3.854275	0.005988	2.063541
Blending Time	26698.16	2	13349.08	1.189023	0.6669	3.340386
Error	2183.401	28	77.9786			
Total	33089.28	44				

Table 4.4 Effect of CRM Binders and Blending Temperature at 30 min on Penetration Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	4422.62	15	315.9015	3.732021	0.001469	2.063541
Blending Temperature	30754.35	2	15377.17	18.66403	9.22E-17	3.340386
Error	2370.094	28	84.64622			
Total	37547.06	44				

Table 4.5 Effect of CRM Binders and Blending Temperature at 60 min on Penetration Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	4379.314	15	312.8081	3.752946	0.00141	2.063541
Blending Temperature	27499.15	2	13749.57	14.96184	0.00003	3.340386
Error	2333.801	28	83.35003			
Total	34212.26	44				

4.3 Analysis of Softening Point Results

Results of the softening point test under different blending temperatures and blending times for various crumb rubber contents are show in Table 4.5 and illustrated in Figures 4.5 – 4.8

Table 4.6 Softening Point Results of Rubberised Bitumen Specimen of Different Blending Conditions

Softening point (°C)							
Mixing time	CRM	160 °C			180 °C		
		Mean	Std. Dev.	n	Mean	Std. Dev.	n
30	0	45.5	0.7	2	47.8	0.4	2
	4	48.8	1.1	2	50.3	0.4	2
	8	51.5	0.0	2	54.3	0.4	2
	12	54.8	0.4	2	56.0	0.0	2
	16	57.8	0.4	2	59.3	0.4	2
	20	61.8	0.4	2	62.0	0.0	2
60	0	46.5	0.7	2	48.3	0.4	2
	4	50.8	1.1	2	51.3	0.4	2
	8	52.5	0.7	2	54.5	0.7	2
	12	55.8	1.1	2	57.5	0.0	2
	16	59.8	1.1	2	60.5	0.0	2
	20	62.8	1.1	2	64.8	0.4	2

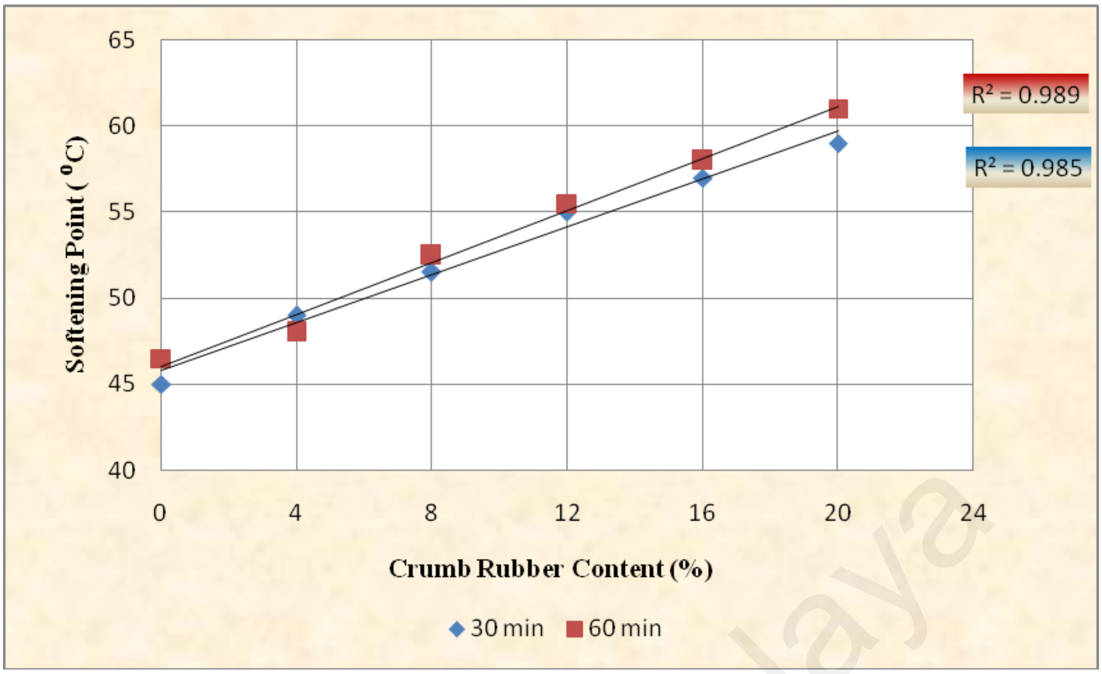


Figure 4.5: Softening Point vs. Blending Time for Specimens with Blending Temperature of 160˚C

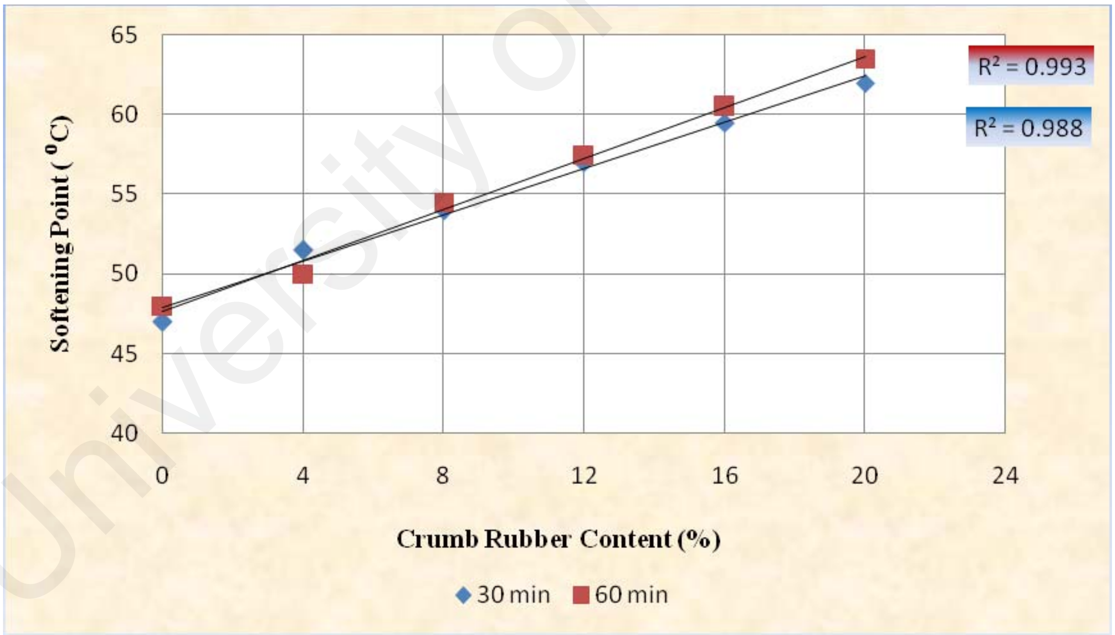


Figure 4.6: Softening Point vs. Blending Time for Specimens with Blending Temperature of 180˚ C

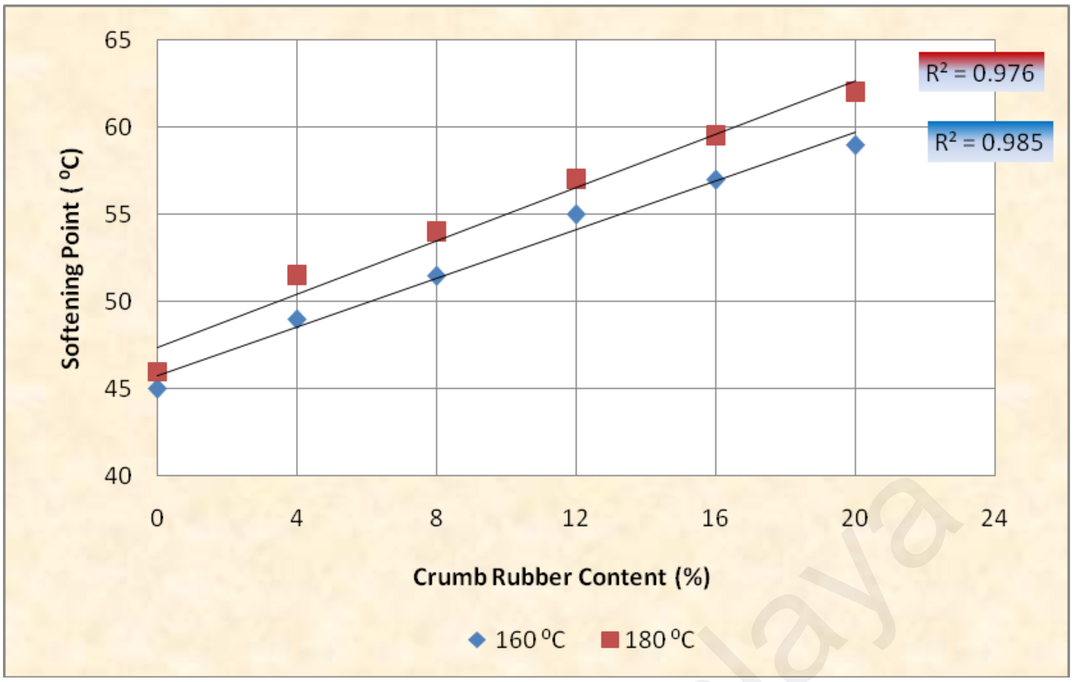


Figure 4.7 : Softening Point vs. Blending Temperature for Specimen with Blending Time of 30 min

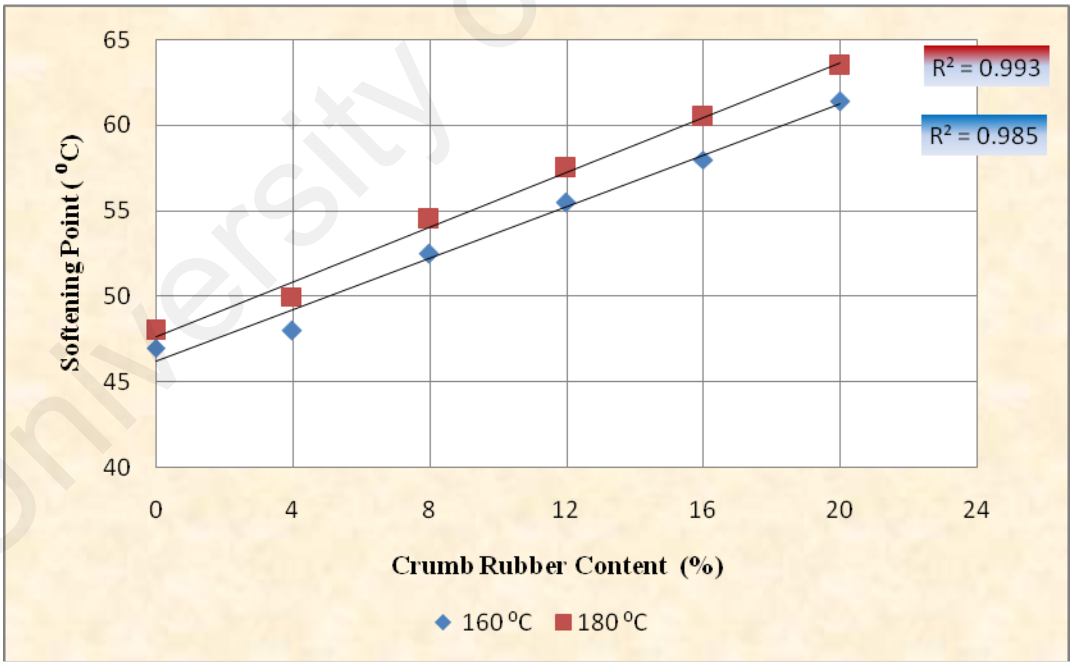


Figure 4.8: Softening Point vs. Blending Temperature for Specimen with Blending Time of 60 min

4.3.1 Effect of Crumb Rubber Content on Softening Point

Figures 4.5- 4.8 show an increase in softening point as the rubber content increased in the bituminous specimens. Figures 4.5 - 4.8 show that the averages increase in softening point of modified binder samples compared with unmodified binder were approximately about 4 to 64 °C for 4 and 20% rubber content respectively. The increase of rubber content in the mix might be related to an increase in the asphaltence/ resins ratio which enhanced the stiffened property and make the modified binder less susceptible to temperature changes.

Statistical analysis of ANOVA indicates a significant difference of crumb rubber content on softening point test for different blending conditions with (p–value) less than the assumed value of 0.05 and the F ratio was more than F critical as displayed in Tables 4.6 -4.9 .

4.3.2 Effect of Blending Time on Softening Point

The variation in softening point values due to varied blending times did not show any significant for all samples (unmodified & modified), for varied mixing temperatures of 160 °C and 180 °C, respectively as shown in Figures 4.5 and 4.6.

The findings showed that there was no significant difference in the softening point results at blending times of 30 min and 60 min respectively, when tested at different temperatures (160 °C and 180 °C).

At blending time of 30 min, as showed in Figure 4.5, the increase in softening point of modified binder samples compared with unmodified binder were approximately 3 °C to 14 °C for crumb rubber content of 4 and 20% respectively. Also, the same trend was obtained at blending time of 60 min; the increase in softening point of modified binder samples compared with unmodified binder were about 1.5 °C to 13.5 °C for 4 and 20% rubber content respectively. Thus, the difference between the two blending time of 30 min and 60 min was about 1.5 °C for all the modified samples. This indicates that the increase in softening point values was highly affected by crumb rubber content rather than the blending time.

Figure 4.6 showed similar findings to Figure 4.5, it shows linear increase in softening point values of rubberised samples with correlation coefficient $R^2 = 0.993$ and 0.988 . The modified bitumen samples of 4% and 20% led to an increase in softening point about 4.5 °C to 10.5 °C at blending time of 30 min, while it showed an increase in softening point about 2 °C to 11.5 °C of rubberised bitumen 4 and 20% respectively. Again the difference between the two blending time was about 1.5 °C for all modified binder mixed at 180 °C.

From statistical analysis of ANOVA, from the comparison of F ratio and F critical, it is evident that blending time has insignificant effect on softening point test for modified and unmodified bitumen samples as illustrated in Tables 4.6 and 4.7 respectively.

4.3.3 Effect of Blending Temperature on Softening Point

The variation in softening point due to varied blending temperature shows significant effect on softening point for all modified samples, while it displayed insignificant influence on the unmodified bitumen for different mixing times of 30 min and 60 min, respectively as shown in Figures 4.7 and 4.8.

Figure 4.7 indicates the effect of different blending temperature at 30 min duration mix. The modified bitumen samples of 4% and 20% led to an increase in softening point of about 4 °C to 14 °C at blending temperature of 160 °C, while it showed an increase in softening point about 5.5 °C to 16 °C of rubberised bitumen with 4 and 20% crumb rubber content respectively.

A similar trend was obtained in Figure 4.8, at 160 °C the increase in CRM content led to high softening point of modified bitumen samples by about 1 °C to 10 °C for 4 and 20% rubber content respectively, while the softening point showed more increase at the blending temperature of 180 °C by about 2.5 °C to 15.5 °C for 4 and 20% crumb rubber content respectively. The increase in blending temperature led to the increase in rubber mass through the interaction and swelling of the rubber into the bitumen during the blending process, thus, leading to increase in the softening point values of rubberised bitumen samples. This increase in softening point was similar with the findings of Bahia and Davis (1994).

According to Liu *et al.* (2009) the main factor in the increase in softening point can be attributed to crumb rubber content, regardless of type and size. The increase in softening point led to a stiff binder that has the ability to enhance its recovery after elastic deformation. Moreover, this increase in softening point might have resulted from the increase in binder molecular weight when the crumb rubber interacted with the bitumen binder. From statistical analysis of ANOVA, it is evident that blending temperature has significant effect on penetration test for modified and unmodified bitumen samples as illustrated in Tables 4.8 and 4.9, respectively. It illustrates that the value of F ratio is higher in comparison to the value F critical and also, the P – value is smaller than ($\alpha = 0.05$).

Table 4.7 Effect of CRM Binders and Blending Time at 160 ° C on Softening Point Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	276.0973	10	30.67748	4.01787	0.005826	2.456281
Blending Time	20530	2	10265	1.420796	0.2455	3.554557
Error	137.4347	18	7.635259			
Total	20943.53	29				

Table 4.8 Effect of CRM Binders and Blending Time at 180 ° C on Softening Point Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	257.3657	10	28.59619	4.009082	0.005891	2.456281
Blending Time	21560.45	2	10780.23	1.348694	0.8893	3.554557
Error	128.3913	18	7.132852			
Total	21946.21	29				

Table 4.9 Effect of CRM Binders and Blending Temperature at 30 min on Softening Point Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	250.6257	10	27.8473	4.031577	0.005727	2.456281
Blending Temperature	20549.21	2	10274.61	14.875	1.03E-20	3.554557
Error	124.3313	18	6.907296			
Total	20924.17	29				

Table 4.10 Effect of CRM Binders and Blending Temperature at 60 min on Softening Point Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	282.104	10	31.34489	4.01206	0.005869	2.456281
Blending Temperature	21578.7	2	10789.35	13.81007	0.00221	3.554557
Error	140.628	18	7.812667			
Total	22001.43	29				

4.4 Analysis of Brookfield Viscosity Results

Results from Brookfield viscosity test at 135 °C under different blending temperatures and blending times for the various crumb rubber contents are shows in Table 4.10 and illustrated in Figures 4.9– 4.12.

Table 4.11: Brookfield Viscosity Results at 135˚C for Different Blending Conditions

Viscosity (MPa- s)							
Mixing time	CRM	160 ºC			180 ºC		
		Mean	Std. Dev.	n	Mean	Std. Dev.	n
30	0	559.3	5.5	3	611.7	6.5	3
	4	1109.3	9.0	3	1793.0	6.6	3
	8	1780.7	4.0	3	2108.3	8.0	3
	12	2588.7	4.5	3	2893.7	6.0	3
	16	3095.7	4.0	3	3294.7	5.0	3
	20	3194.7	5.0	3	3772.3	6.1	3
60	0	550.7	3.1	3	620.7	6.1	3
	4	1451.3	4.2	3	1901.3	7.1	3
	8	2009.7	9.5	3	2175.3	5.0	3
	12	2766.7	4.2	3	2992.0	6.6	3
	16	3211.3	8.1	3	3383.0	5.6	3
	20	3494.7	6.1	3	3985.7	4.5	3

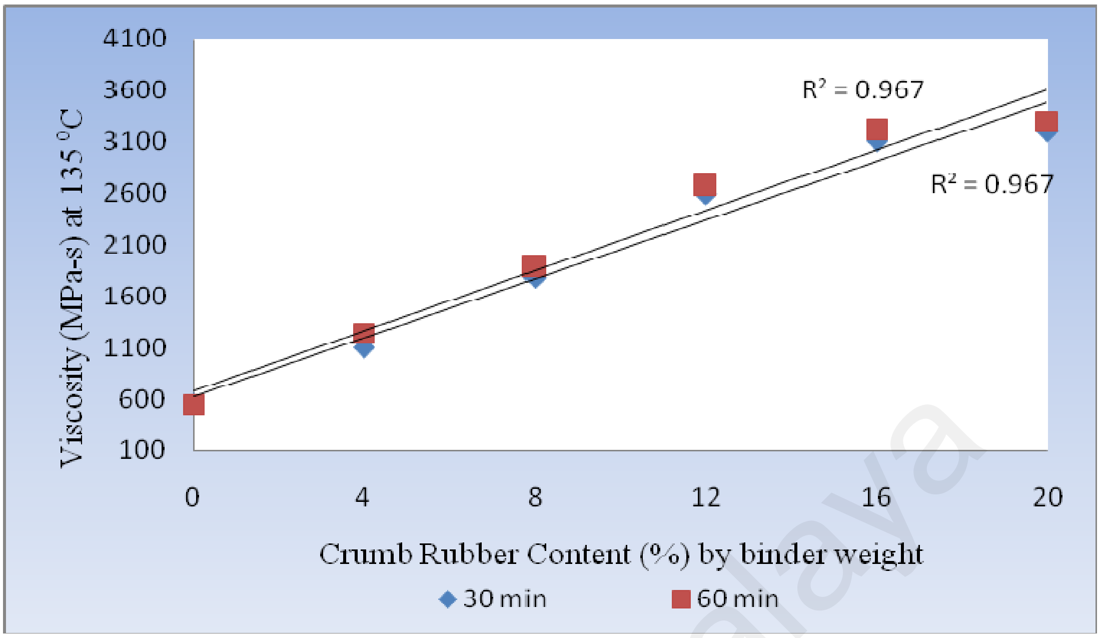


Figure 4.9: Brookfield Viscosity at 135˚C vs. Blending Time for Specimens with Blending Temperature of 160˚C

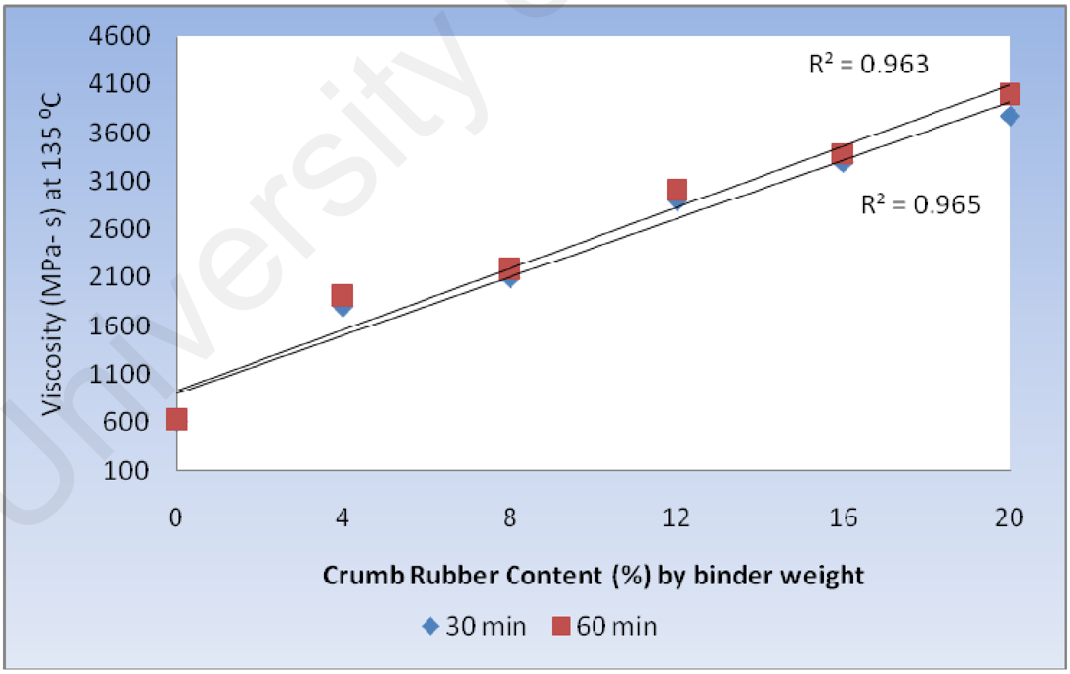


Figure 4.10: Brookfield Viscosity at 135˚C vs. Blending Time for Specimens with Blending Temperature of 180˚C.

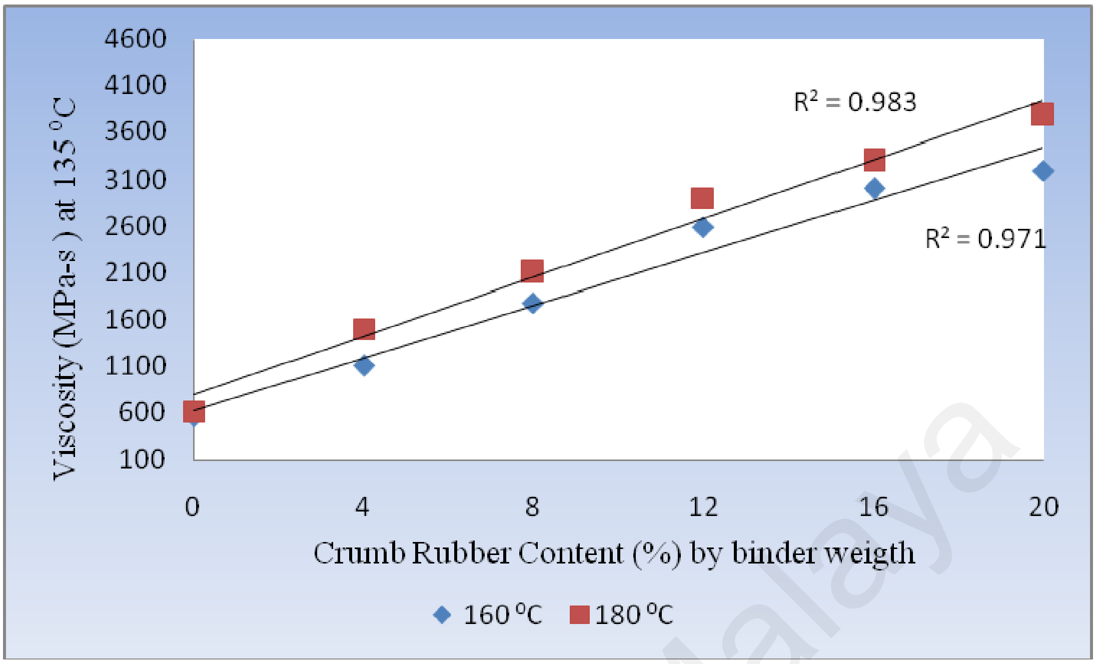


Figure 4.11: Brookfield Viscosity at 135˚C vs. Blending Temperatures for Specimens with Blending Time of 30 min

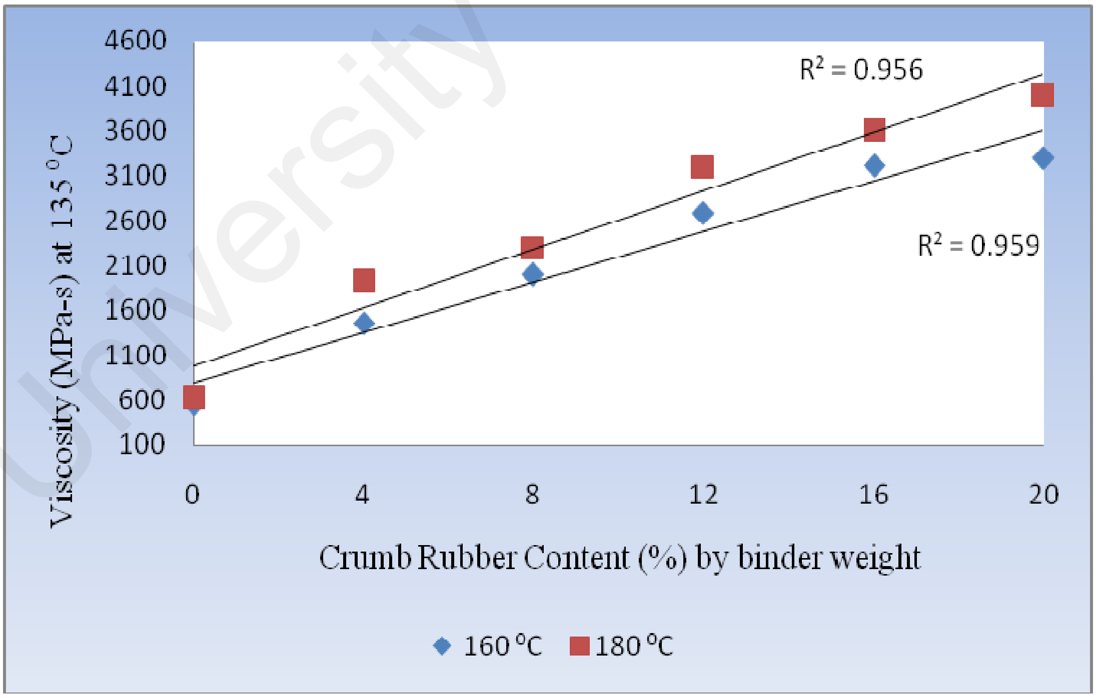


Figure 4.12: Brookfield Viscosity at 135˚C vs. Blending Temperatures for Specimen with Blending Time of 60 min

4.4.1 Effect of Crumb Rubber Content on Viscosity

Figures 4.9- 4.12 showed an increase in Brookfield viscosity as the rubber content was increased in the bituminous specimens. The increase is linearly dependent with correlation factor about 0.965 – 0.983 for all the figures. From Figures 4.9- 4.12 , it is evident that the averages increase in viscosity of modified binder samples compared with unmodified binder were approximately about 25 to 80% for 4 and 20% rubber content respectively.

The increase in viscosity might be due to the amount of asphaltense in the bitumen that enhanced the viscous flow of the modified bitumen sample during the interaction process. In general, higher crumb rubber content was found to lead to an increase in the viscosity at 135 °C (Jeong *et al.*, 2010). According to The Asphalt Institute (2003), the specification of a maximum viscosity at 135 °C should not be more than 3Pas for unaged binder. Thus, the viscosity of 16% and 20% rubber content at different conditions of time and temperature has exceeded the limit and thus, it is not recommended.

Statistical analysis of ANOVA indicates a significant difference of crumb rubber content on viscosity test for different blending conditions with (p–value) less than the assumed value of 0.05 and the F ratio was more than F critical as displayed in Tables 4.11 - 4.14.

4.4.2 Effect of Blending Time on Brookfield Viscosity

The variation in viscosity values due to varied blending times did not show any significant effect on viscosity for all samples (unmodified & modified), for varied mixing temperatures of 160 °C and 180 °C, respectively as shown in Figures 4.9 and

4.10. The results showed that there were no significant differences in the viscosity at blending times of 30 min and 60 min respectively, when tested at different temperatures (160 °C and 180 °C).

From statistic analysis ANOVA, by comparing between F ratio and F critical, it is evident that blending time has an insignificant effect on viscosity test for modified and unmodified bitumen samples as illustrated in Tables 4.11 and 4.12, respectively.

4.4.3 Effect of Blending Temperature on Brookfield Viscosity

The variation in viscosity due to varied blending temperature showed significant effect for all modified samples, while it displayed insignificant influence on the unmodified bitumen for different mixing times of 30 min and 60 min, respectively as shown in Figures 4.11 and 4.12.

The increase in viscosity with increased blending conditions (blending time and blending temperature) has been verified by numerous researchers (Bahia and Davies 1995b; Billiter *et al.* 1996; Lee *et al.*, 2006; Jeong *et al.* 2010). Based on the viscosity results of this study, its increase was accompanied by the increase in rubber content from 4% to a high CRM content of 20% by binder weight. Such results were similar to the findings in previous research as well (Jeong *et al.*, 2010 ; Rosner and Chehovits ,1982).

From statistical analysis of ANOVA, it is evident that blending temperature has significant effect on viscosity of modified and unmodified bitumen samples as illustrated in Tables 4.13 and 4.14 respectively. The value of F ratio is higher in comparison to the value F critical and also, the P – value is smaller than ($\alpha = 0.05$).

Based on the results, it can be concluded that the blending conditions (crumb rubber content, blending temperature and blending time) play together to improve the viscosity at 135 °C. This increase might be due to CRM content with higher blending temperature, which would increase the elasticity and break down the crosslink of rubber; these aspects would make the modified binder thicker and more elastic. In addition, during sample preparation it is highly important to select the temperature at which the binder would maintain an acceptable viscosity that enables the bitumen binder to coat the aggregate effectively. This in turn would, help to ensure better rubberised bitumen on permanent deformations.

The results of Tables 4.15 – 4.20 show that the viscosity decreases as the temperature of rubberised bitumen binder increases at different temperatures (90 °C, 110 °C, 120 °C, 135 °C, 150 °C and 170 °C). The crumb rubber-modified bitumen binder exhibited higher viscosities compared with the unmodified bitumen. The results showed that the viscosity of the samples decreased as the temperature increase. This indicates that temperature has a direct effect on the viscosity of the modified samples as the results displayed a rapid decrease at temperatures of 90 °C to 135 °C.

Table 4.12: Effect of CRM Binders and Blending Time at 160 ^{o} C on Viscosity Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	12085439	15	863245.7	3.967917	0.000934	2.063541
Blending Time	61423180	2	30711590	1.16613	0.23887	3.340386
Error	6091578	28	217556.4			
Total	79600197	44				

Table 4.13: Effect of CRM Binders and Blending Time at 180 ^{o} C on Viscosity Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	11247541	15	803395.8	3.98694	0.000901	2.063541
Blending Time	80177738	2	40088869	1.945418	0.228665	3.340386
Error	5642192	28	201506.9			
Total	97067471	44				

Table 4.14: Effect of CRM Binders and Blending Temperature at 30 min on Viscosity Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	11590713	15	827908.1	3.83675	0.0012	2.063541
Blending Temperature	67002860	2	33501430	15.2547	0.0001	3.340386
Error	6041943	28	215783.7			
Total	84635516	44				

Table 4.15: Effect of CRM Binders and Blending Temperature at 60 min on Viscosity Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	11524269	15	823162	3.900036	0.001063	2.063541
Blending Temperature	75588899	2	37794450	17.06527	1.11E-16	3.340386
Error	5909826	28	211065.2			
Total	93022994	44				

Table 4.16: Brookfield Viscosity Results of unmodified bitumen

	Viscosity (MPa –s)						
	Temperatures (^{o}C)						
	90	**100**	**120**	**135**	**150**	**160**	**170**
Blending mix 160˚C , 30min	7300	2970	670	560	360	275	95
Blending mix 160˚C, 60min	7340	3200	750	550	389	289	100
Blending mix 180˚C,30min	8700	3450	840	611	422	344	145
Blending mix 180˚C,60min	9600	3880	920	620	478	388	189

Table 4.17: Brookfield Viscosity Results of 4% CRM modified bitumen

	Viscosity (MPa –s)						
	Temperatures (^{o}C)						
	90	**100**	**120**	**135**	**150**	**160**	**170**
Blending mix 160˚C , 30min	9537	3975	1304	1100	510	200	120
Blending mix 160˚C, 60min	10875	4950	1500	1450	430	254	137
Blending mix 180˚C,30min	10012	3817	1887	1800	310	300	224
Blending mix 180˚C,60min	11065	4882	1930	1909	250	325	250

Table 4.18: Brookfield Viscosity Results of 8 % CRM modified bitumen

	Viscosity (MPa –s)						
	Temperatures (^{o}C)						
	90	**100**	**120**	**135**	**150**	**160**	**170**
Blending mix 160˚C , 30min	10354	4841	3780	1780	250	220	135
Blending mix 160˚C, 60min	10540	5000	4100	2000	325	310	220
Blending mix 180˚C,30m	10711	5824	4400	2100	465	350	310
Blending mix 180˚C,60min	13094	6055	2200	2183	683	411	322

Table 4.19: Brookfield Viscosity Results of 12 % CRM Modified Bitumen

	Viscosity (MPa –s)						
	Temperatures (°C)						
	90	**100**	**120**	**135**	**150**	**160**	**170**
Blending mix 160˚C , 30min	9555	5691	4670	2589	400	280	153
Blending mix 160˚C, 60min	9900	8600	3888	2770	417	290	174
Blending mix 180˚C,30min	10450	5692	3340	2900	562	325	233
Blending mix 180˚C,60min	11200	8800	3100	2999	1090	637	388

Table 4.20: Brookfield Viscosity Results of 16 % CRM Modified Bitumen

	Viscosity (MPa –s)						
	Temperatures (°C)						
	90	**100**	**120**	**135**	**150**	**160**	**170**
Blending mix 160˚C , 30min	11300	10100	4200	3100	810	422	210
Blending mix 160˚C, 60min	11890	10900	4780	3210	854	530	270
Blending mix 180˚C,30min	12450	11200	4995	3300	890	617	330
Blending mix 180˚C,60min	13111	12200	5100	3386	930	679	388

Table 4.21: Brookfield Viscosity Results of 20% CRM Modified Bitumen

	Viscosity (MPa –s)						
	Temperatures (°C)						
	90	**100**	**120**	**135**	**150**	**160**	**170**
Blending mix 160˚C , 30min	10450	9880	4769	3200	617	325	220
Blending mix 160˚C, 60min	10900	10440	4900	3500	788	469	278
Blending mix 180˚C,30m	12300	11567	5200	3779	1250	590	340
Blending mix 180˚C,60min	13450	12780	5889	3990	1875	799	390

4.5 Analysis of Ductility Results

The ductility results under different blending temperatures and blending times for various crumb rubber contents are shown in Table 4.21 and illustrated in Figures 4.13–4.16.

Table 4.22: Ductility Results of CRM Binders at 25 ˚C of Different Blending Conditions

Ductility (cm)							
Mixing time	CRM	160 °C			180 °C		
		Mean	Std. Dev.	n	Mean	Std. Dev.	n
30	0	105.0	7.1	2	93.0	2.8	2
	4	93.0	4.2	2	82.5	3.5	2
	8	72.5	3.5	2	73.5	3.5	2
	12	54.0	4.2	2	63.5	2.1	2
	16	48.0	2.8	2	48.5	2.1	2
	20	43.5	2.1	2	40.0	4.2	2
60	0	99.0	1.4	2	91.5	3.5	2
	4	82.5	3.5	2	73.5	4.9	2
	8	62.5	2.1	2	59.5	4.9	2
	12	52.5	2.1	2	47.0	4.2	2
	16	49.5	2.1	2	40.5	4.9	2
	20	41.0	4.2	2	31.0	5.7	2

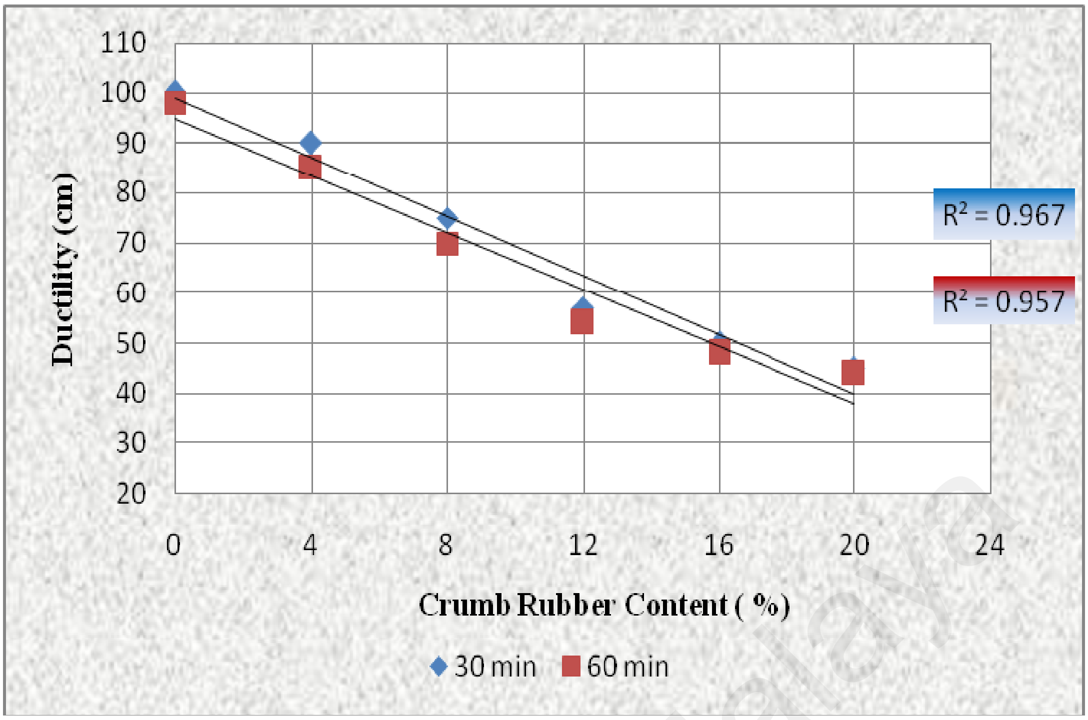


Figure 4.13: Ductility vs. Blending Time for Specimens with Blending Temperature of 160 °C

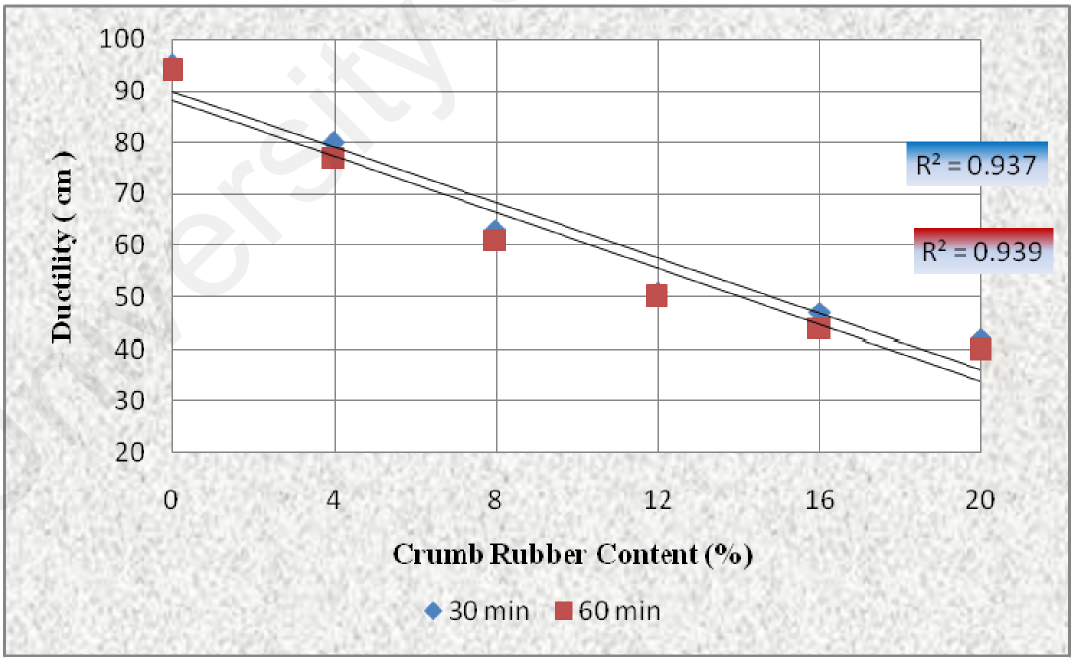


Figure 4.14: Ductility vs. Blending Time for Specimens with Blending Temperature of 180 °C

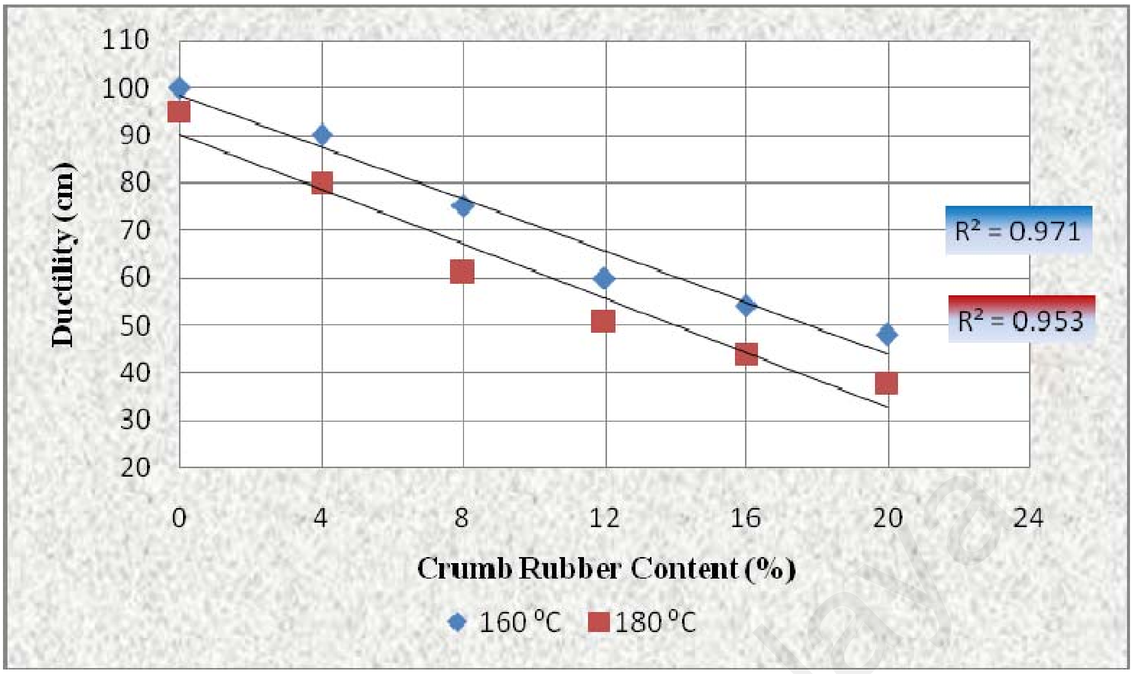


Figure 4.15: Ductility vs. Blending Temperatures for specimens with Blending Time of 30 min

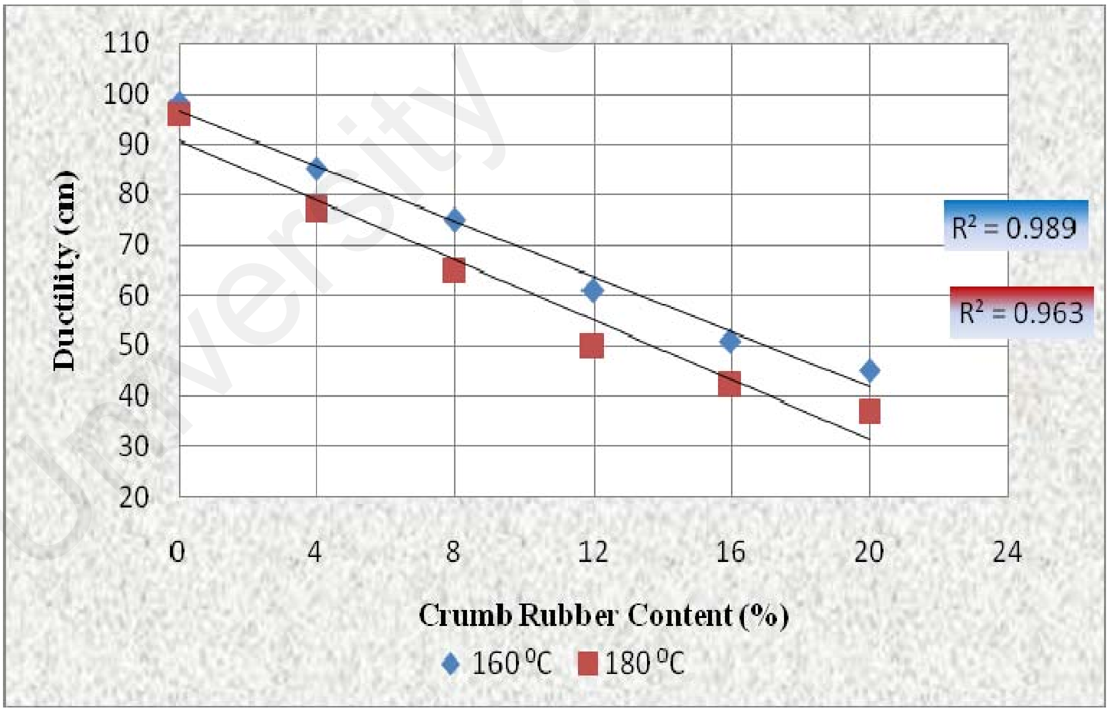


Figure 4.16: Ductility vs. Blending Temperatures for specimens with Blending Time of 60 min

4.5.1 Effect of Crumb Rubber Content on Ductility

Figures 4.13 - 4.16 show that ductility decreased dramatically as the rubber content was increased in the bituminous specimens. From Figures 4.13- 4.16, the average decrease in ductility of modified binder samples compared with unmodified binder were approximately about 15 to 89 % for 4 and 20% rubber content respectively.

Statistical analysis of ANOVA indicates a significant difference of crumb rubber content on ductility test for different blending conditions with (p–value) less than the assumed value of (0.05) and the F ratio was more than the F critical as displayed in Tables 4.22 -4.25.

The results could be explained by the physical and chemical interactions during the blending process of the rubberised bitumen. Accordingly, an increase in binder mass could make the binder more elastic, stiff and highly resistant to pavement rutting.

Meanwhile, the decrease in ductility value could be attributed to the oily part of the bitumen being absorbed into the rubber powder and the increase in mass of the rubber particles. In effect, the modified binder became thicker compared with the unmodified samples.

4.5.2 Effect of Blending Time on Ductility

The variation in ductility values due to varied blending times did not show any significant effect on ductility properties for all samples (unmodified & modified), for varied mixing temperatures of 160 °C and 180 °C, respectively as shown in Figures 4.13 and 4.14.

Figures 4.13 and 4.14 show that the crumb rubber content has an obvious effect on ductility as it decreased from 4% to 20% rubber content, whereas the blending time showed insignificant effect on ductility values. The ductility results of Figure 4.13 shows an average difference of rubberised bitumen binders compared with the unmodified bitumen of about 15 cm (4% rubber content) and 49 cm (20% rubber content). Longer blending time of (60 min) decreased the ductility to about 40 cm (20% rubber content), as shown in Figure 4.14.

From statistic analysis ANOVA, it is evident that blending time has insignificant effect on ductility test for modified and unmodified bitumen samples as illustrated in Tables 4.22 and 4.23 respectively.

4.5.3 Effect of Blending Temperature on Ductility

The variation in ductility values due to varied blending temperatures show a significant effect for all modified samples. On the other hand, it displayed insignificant influence on unmodified bitumen for different mixing times of 30 min and 60 min, respectively as shown in Figures 4.15 and 4.16.

A higher blending temperature of rubberised bitumen binder led to better ductility results for samples of 4 % – 20% rubber content by about 11.1 – 20.9% at 30 minutes blending time with correlation coefficient ($R^2 = 0.97$ and 0.95) and about 19.5 – 48.8% at 60 minutes blending time with correlation coefficient $R^2 = 0.98$ and 0.96, respectively as shows in Figures 4.15 and 4.16. From statistical analysis of ANOVA, it is evident that blending temperature has significant effect on ductility test for modified and unmodified bitumen samples as illustrated in Tables 4.24 and 4.25 respectively. The value of F ratio is higher in comparison to the value F critical and also, the P – value is smaller than ($\alpha = 0.05$).

As indicated by the ductility results, the sample of rubberised bitumen exhibited less susceptibility to high temperature changes at blending conditions of 180 °C and 60 min in relation to the 4% - 20% crumb rubber contents. The ductility result of this study was consistent with the results of the penetration, softening point and viscosity tests. The addition of high content of crumb rubber improved the physical properties of rubberised bitumen by increasing the ductility and elastic recovery. Thus, the modified binder could exhibit behaviours that are more elastic, eventually leading to better pavement performance (Becker *et a*l., 2001).

Table 4.23: Effect of CRM Binders and Blending Time at 160 °C on Ductility Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	3528.837	10	392.093	3.628261	0.009598	2.456281
Blending Time	23930.12	2	11965.06	0.719563	0.7774	3.554557
Error	1945.195	18	108.0664			
Total	29404.15	29				

Table 4.24: Effect of CRM Binders and Blending Time at 180 °C on Ductility Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	3098.231	10	344.2479	3.59778	0.009991	2.456281
Blending Time	21418.38	2	10709.19	1.923146	0.7111	3.554557
Error	1722.301	18	95.68341			
Total	26238.91	29				

Table 4.25: Effect of CRM Binders and Blending Temperature at 30 min on Ductility Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	3751.424	10	416.8249	3.491958	0.011495	2.456281
Blending Temperature	25446.92	2	12723.46	10.59101	0.0001	3.554557
Error	2148.608	18	119.3671			
Total	31346.95	29				

Table 4.26: Effect of CRM Binders and Blending Temperature at 60 min on Ductility Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	2873.644	10	319.2938	3.778903	0.007891	2.456281
Blending Temperature	19584.24	2	9792.121	11.89163	0.000011	3.554557
Error	1520.888	18	84.49378			
Total	23978.77	29				

4.6 Analysis of Elastic Recovery Results

The elastic recovery test results under different blending temperatures and blending times for various crumb rubber contents are shows in Table 4.26 and illustrated in Figures 4.17– 4.20.

Table 4.27: Elastic Recovery Results of CRM Binders 25˚C for Different Blending Conditions

Elastic recovery (%)							
Mixing time	CRM	160 °C			180 °C		
		Mean	Std. Dev.	n	Mean	Std. Dev.	n
30	0	13.0	1.4	2	17.5	0.7	2
	4	52.0	1.4	2	63.5	3.5	2
	8	67.0	2.8	2	74.0	2.8	2
	12	77.0	4.2	2	82.0	2.8	2
	16	87.0	2.8	2	90.3	0.4	2
	20	93.0	1.4	2	95.8	1.1	2
60	0	16.0	1.4	2	19.0	1.4	2
	4	55.5	2.1	2	66.0	4.2	2
	8	72.5	3.5	2	75.5	3.5	2
	12	79.0	2.8	2	85.5	3.5	2
	16	89.0	1.4	2	92.3	1.1	2
	20	94.0	1.4	2	95.5	0.7	2

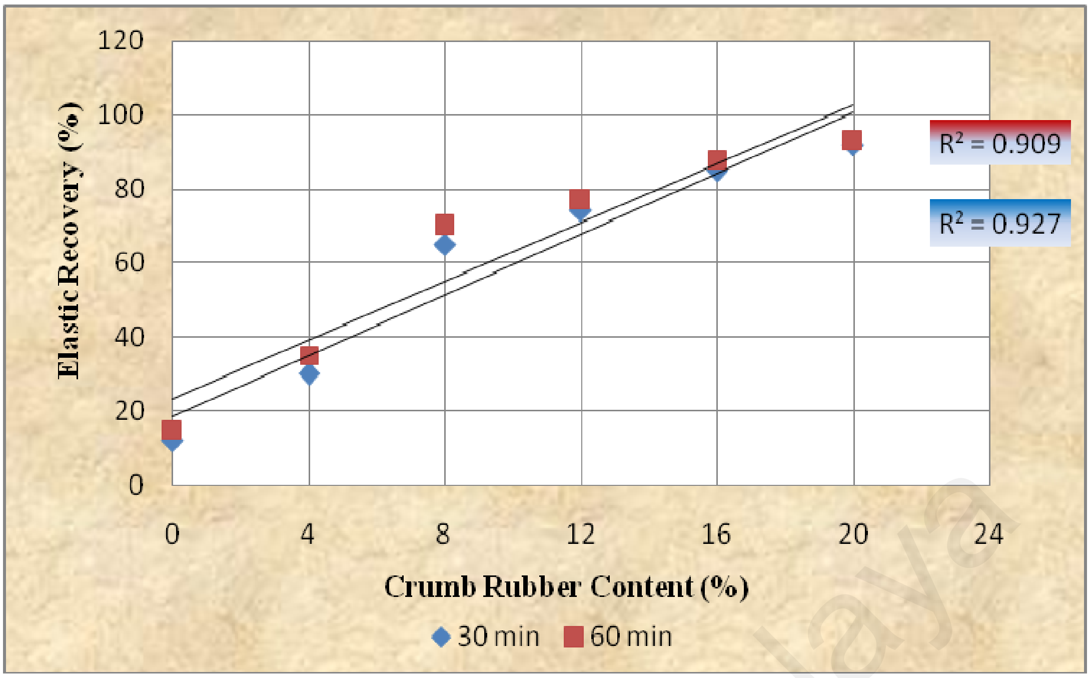


Figure 4.17: Elastic Recovery vs. Blending Time for Specimens with Blending Temperature of 160 °C

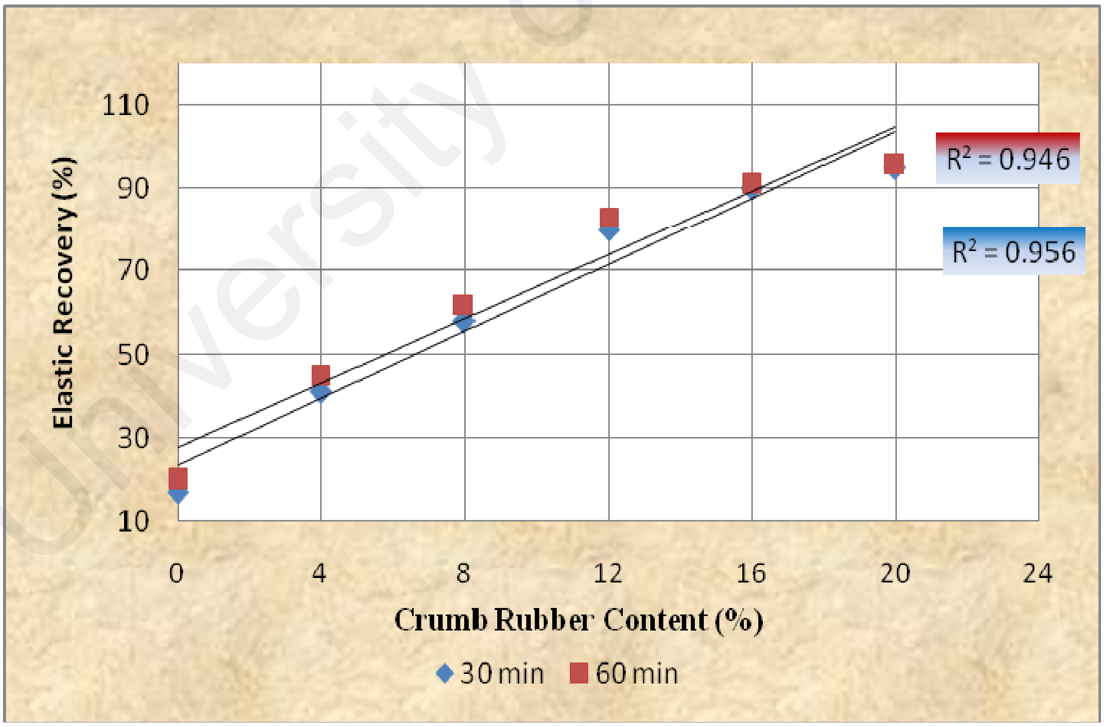


Figure 4.18: Elastic Recovery vs. Blending Time for Specimens with Blending Temperature of 180 °C

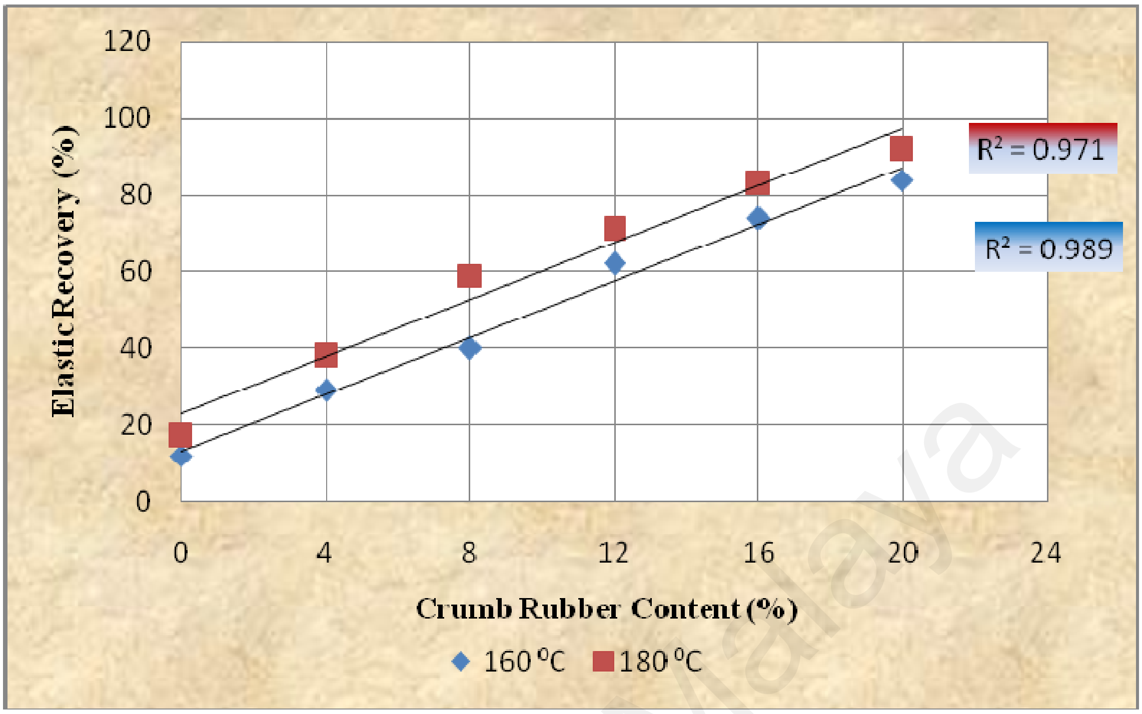


Figure 4.19: Elastic Recover vs. Blending Temperature for Specimens with Blending Time of 30 min

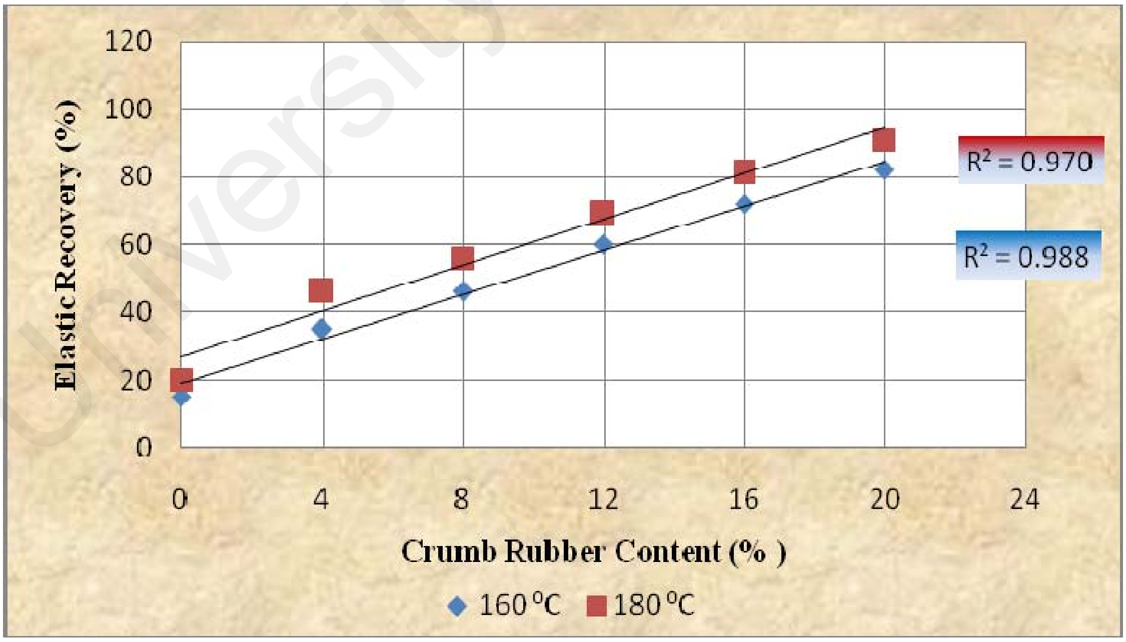


Figure 4.20: Elastic Recover vs. Blending Temperature for Specimens with Blending Time of 60 min

4.6.1 Effect of Crumb Rubber Content on Elastic Recovery

As displayed in Figures 4.17- 4.20, an increase in crumb rubber content (CRM) causes the degree of elastic recovery to increase dramatically and linearly with strong correlation coefficient $R^2 > 0.93$ for all modified bitumen samples regardless of different blending conditions.

From Figures 4.17- 4.20, the increase in elastic recovery of modified binder samples compared with unmodified binder were approximately about 14 to 91% for 4 and 20% rubber content respectively.

Elastic recovery results were similar to ductility results of rubberised bitumen as it shows significant effect of high content of crumb rubber on elasticity of the modified binder, which presented consistency with binder elasticity and recovery after deformation, hence improving binder resistance to rutting. The elastic recovery values increased from 16.38% for the unmodified binder to 94.5% for the 20% crumb rubber content by binder weight, indicating the ability of the binder to recover its shape after removing the strain load. In general, the crumb rubber consists of elastomers (natural and synthetic rubber); it is well known that synthetic rubber increases the elastic behaviour of the bitumen modified binder, while the use of natural rubber leads to an increase in thermal behaviour (Memon and Chollar, 1997).

The increase in rubber content leads to increase in elastic recovery results and it is similar to ductility results of rubberised bitumen, which presented consistency with binder elasticity, rheology, rutting resistance and elastic recovery after deformation. Hence, lead to improving modified binder's resistance to rutting.

Bahia *et al.* (1998) compared five modified binders by testing elastic recovery, ductility and resilience. The results of their study showed that these conventional measurements were inconsistent in ranking in terms of suitability for the polymer-modified binders. Oliver (1997) who examined rheology property of mixes and their effect on rutting by using the wheel-tracking test reported that no relationship exists between rut resistance and elastic recovery or softening point.

Statistical analysis of ANOVA indicates a significant difference of crumb rubber content on elastic recovery for different blending conditions with (p–value) less than the assumed value of 0.05 and the F ratio was more than the F critical as displayed in Tables 4.27 - 4.30.

4.6.2 Effect of Blending Time on Elastic Recovery

The variation in elastic recovery values due to varied blending times did not show any significant improvement for all samples (unmodified & modified), for varied mixing temperatures of 160 °C and 180 °C, respectively as shown in Figures 4.17 and 4.18. A longer blending time of 60 minutes exhibited slightly different findings compared with blending time of 30 minutes under different blending temperatures, meaning that the two blending time (30 and 60 min) have similar effect on modified binder properties. These results are similar to the viscosity, penetration, softening point and ductility results, which show slight difference of blending time effect on modified bitumen binder at increased blending times of 30–60 min.

From statistical analysis of ANOVA, it is evident that blending time has insignificant effect on elastic recovery for modified and unmodified bitumen samples as illustrated in Tables 4.27 and 4.28 respectively.

4.6.3 Effect of Blending Temperature on Elastic Recovery

From Figures 4.19 and 4.20, an increasing effect was evident with the elastic recovery for all modified bitumen samples at 160 ºC and 180 ºC respectively. However, for unmodified bitumen samples there was insignificant influence on the elastic recovery.

Higher blending temperature affected the elastic recovery of modified bitumen binders (Figures 4.19 and 4.20). As shown in Figure 4.19, the increase in elastic recovery of modified bitumen of 4% rubber content was about 54% - 63% as blending temperature increased from160 °C to 180 °C respectively. Also, rubberised samples of 20% rubber content resulted in an increase in elastic recovery by about 89% - 96% when blending temperatures increased from 160 °C to 180 °C at 60 minutes blending time. It seems that higher blending temperature has significant effect on the elastic recovery of the modified bitumen by increasing the rubber mass due to the absorption of maltense from the bitumen binder through the interaction of rubber- bitumen. Thus, the modified binder become more elastic and thus improved its resistance to elastic deformation under high tensile stress.

From statistical analysis of ANOVA, it is evident that blending temperature has significant effect on elastic recovery for modified and unmodified bitumen samples as illustrated in Tables 4.29 and 4.30 respectively. It illustrates that the value of F ratio is higher in comparison to the value F critical and also, the P – value is smaller than (α = 0.05).

Table 4.28: Effect of CRM Binders and Blending Time at 160 °C on Elastic Recovery Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	2684.304	10	298.256	4.002457	0.00594	2.456281
Blending Time	39033.8	2	19516.9	2.907769	0.49431	3.554557
Error	1341.328	18	74.51822			
Total	43059.43	29				

Table 4.29: Effect of CRM Binders and Blending Time at 180 °C on Elastic Recovery Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	1721.266	10	191.2517	4.010445	0.005881	2.456281
Blending Time	44739.97	2	22369.99	2.086463	0.299758	3.554557
Error	858.3913	18	47.68841			
Total	47319.63	29				

Table 4.30: Effect of CRM Binders and Blending Time at 30 min on Elastic Recovery Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	2315.957	10	257.3286	3.859689	0.007115	2.456281
Blending Temperature	40765.26	2	20382.63	5.720397	0.0001	3.554557
Error	1200.075	18	66.67081			
Total	44281.29	29				

Table 4.31: Effect of CRM Binders and Blending Time at 60 min on Elastic Recovery Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	2030.779	10	225.6421	3.837168	0.007323	2.456281
Blending Temperature	43168.69	2	21584.34	7.053613	0.000015	3.554557
Error	1058.478	18	58.80433			
Total	46257.94	29				

4.7 Analysis of Dynamic Shear Rheometer Test Results

Dynamic shear rheometer (DSR) was used to measure and determine the rheological properties of the bitumen binder. Bitumen is known as a colloidal system with high molecular weight components (asphaltnese) and low molecular weight maltenes. Upon reaching the level of maltenes and asphaltene concentrations, the bitumen could move either in solid or gel type. Colloidal systems could reflect the rheological properties of bitumen binders (Teugels, 2000). DSR results included parameters of complex shear modulus (G*), phase angle (δ), storage modulus (G'), loss modulus (G'') and stiffness (G*/sin δ).

4.7.1 Results and Analysis of Complex shear modulus (G*) at 76 °C

The test results of complex shear modulus G* at 76 °C under different blending temperatures and blending times for various crumb rubber contents are shown in Table 4.31 and has illustrated in Figures 4.21–4.24.

Table: 4.32: Complex Shear Modulus for Different Blending Conditions at 76˚C

Complex shear modulus G* (MPa)							
Mixing time	CRM	160 °C			180 °C		
		Mean	Std. Dev.	n	Mean	Std. Dev.	n
30	0	232.0	2.6	3	306.0	2.0	3
	4	754.3	2.1	3	1002.3	3.2	3
	8	1316.3	3.1	3	1606.0	5.3	3
	12	1875.7	3.5	3	2016.0	4.6	3
	16	2130.7	4.0	3	2229.7	2.5	3
	20	2423.0	3.0	3	2405.0	4.6	3
60	0	293.7	3.5	3	262.0	2.0	3
	4	885.7	3.5	3	1042.3	5.4	3
	8	1452.3	2.5	3	1842.0	3.0	3
	12	1924.3	4.0	3	2073.0	4.0	3
	16	2181.0	4.0	3	2291.7	2.5	3
	20	2461.0	5.6	3	2565.0	4.6	3

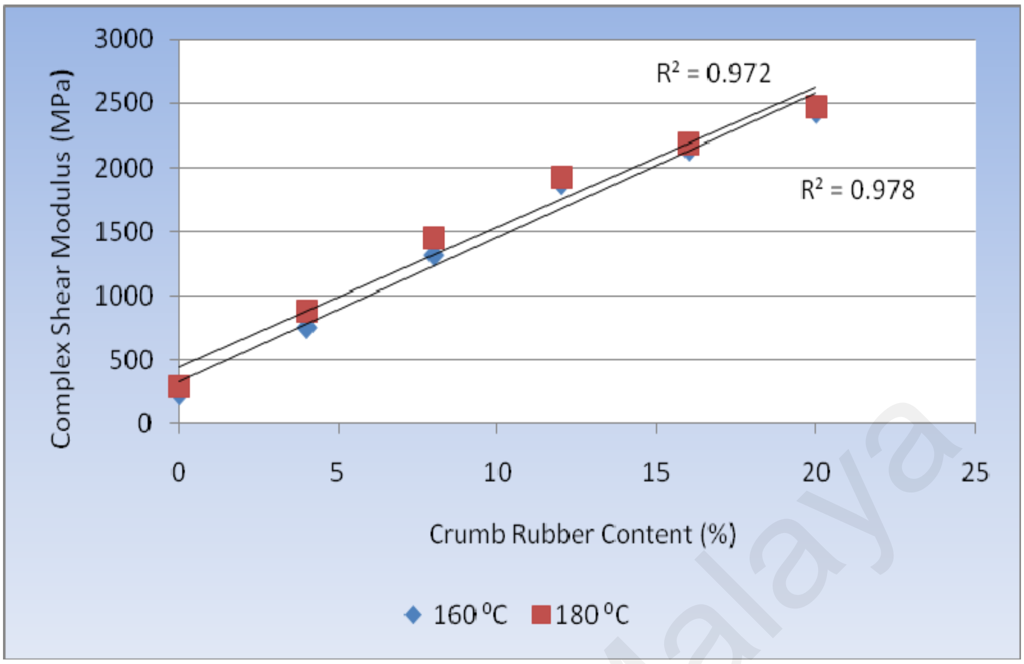


Figure 4.21: Complex Shear Modulus at 76 °C vs. Blending time for Specimens with Blending Temperature of 160 °C

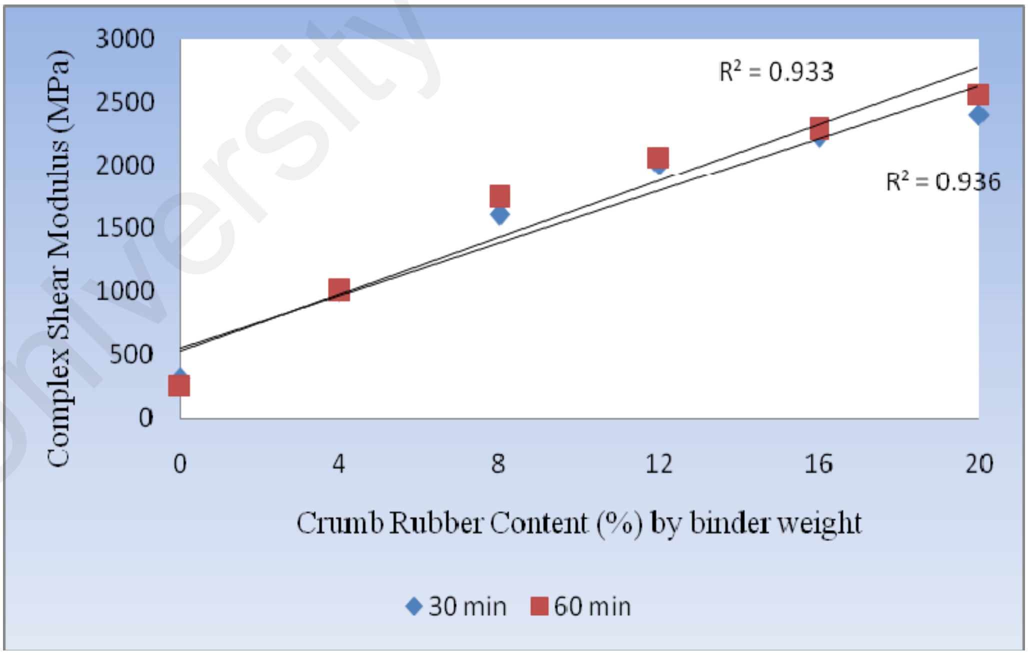


Figure 4.22: Complex Shear Modulus at 76 °C vs. Blending time for Specimens with Blending Temperature of 180 °C

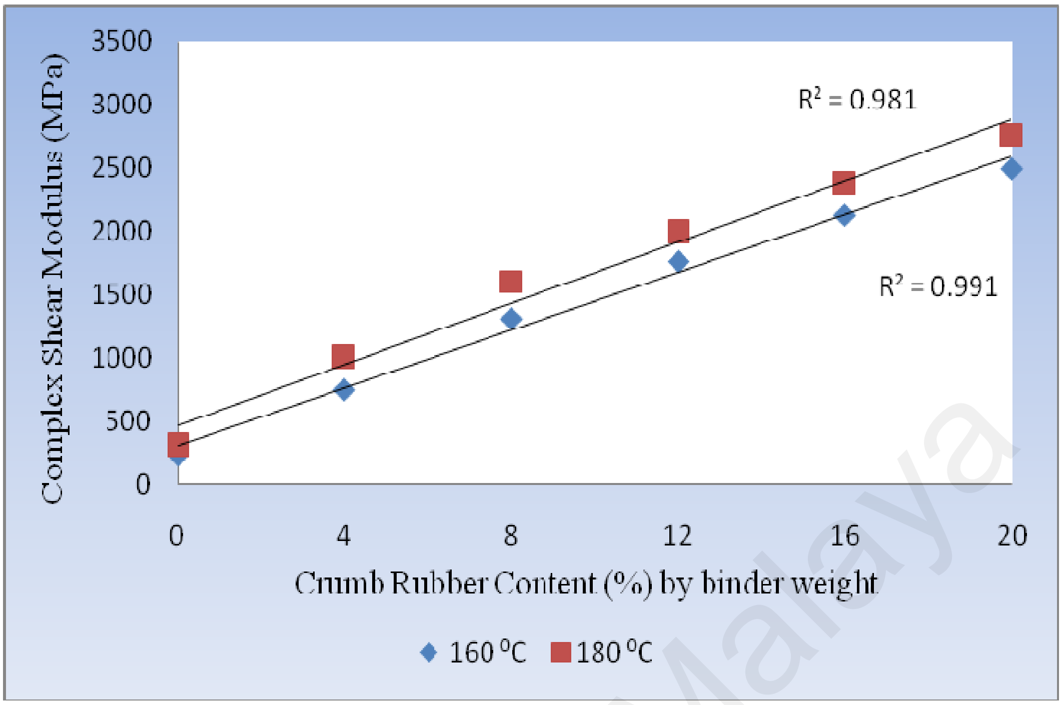


Figure 4.23: Complex Shear Modulus at 76 °C vs. Blending Temperature for Specimen with Blending Time of 30 min

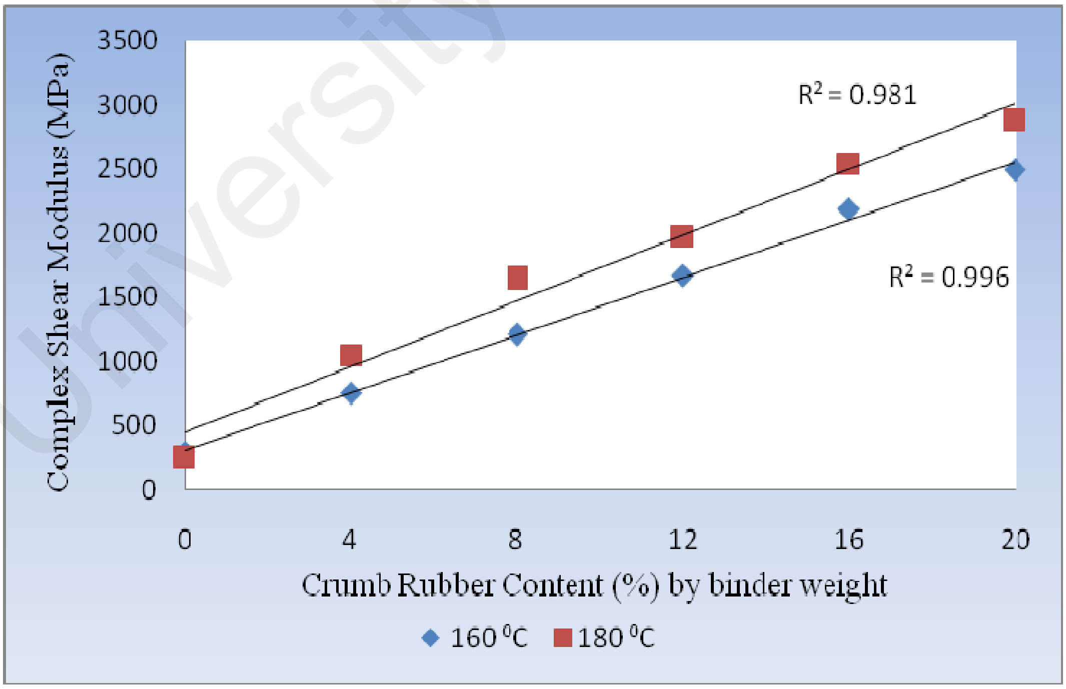


Figure 4.24: Complex Shear Modulus at 76 °C vs. Blending Temperature for Specimen with Blending Time of 60 min

4.7.1.1 Effect of Crumb Rubber Content on G* at 76 °C

Figures 4.21 – 4.24 showed increase in complex shear modulus as the crumb rubber content increases in the bituminous samples. The increase is linearly dependent with correlation factors R^2 were about 0.933- 0.996. Furthermore, the crumb rubber content exhibited a strong effect on complex shear modulus improvement by increasing the stiffness of crumb rubber modified bitumen binder.

4.7.1.2 Effect of Blending Time on G* at 76 °C

The variation in complex shear modulus due to varied blending times did not show any significant effect on G* for unmodified bitumen samples and rubberised bitumen, for varied mixing temperatures of 160 °C and 180 °C, respectively as shown in Figures 4.21- 4.22. The increase is linearly dependent with correlation factor equal to 0.97 for all the figures. The findings showed that there was no significant difference in G* results at blending times 30 min and 60 min respectively, when tested at different temperatures (160 °C and 180 °C).

Figures 4.21 and 4.22 show that the effect of crumb rubber content has an obvious effect on complex shear modulus (G*) as it increased from 4% to 20% rubber content. Whereas the blending time showed insignificant effect on G* values. The complex shear modulus results of Figure 4.21 show average increase of rubberised bitumen binders compared with the unmodified bitumen, at blending time of 30 min which was about 5.9 and 25.5 for samples of 4% and 20% rubber content respectively. In the case of Figure 4.22, the average increase in G*, at blending time 30 min, was about 9 to 26 for samples of 4% and 20% rubber content .

The results indicate the average increase of G* of modified binder compared to unmodified binder at 30 and 60 min had no effect on binder shear modulus as shown in figures 4.21 and 4.22.

Tables 4.32 and Table 4.33 show the results of ANOVA ($\alpha = 0.05$) of the complex shear modulus results. Evidently, there were no significant differences for blending time on G* for samples of modified and unmodified binders. Tables 4.6.1 and Table 4.6.2 show that the crumb rubber contents seems to have a significant difference on G* results as the p-value was less than the assumed value of 0.05 of about 0.0007 and 0.001 respectively.

4.7.1.3 Effect of Blending Temperature on G* at 76 °C

Blending temperature presented a significant effect in terms of improving the stiffness G* as shown in Figures 4.23 and 4.24. A higher blending temperature of rubberised bitumen binder led to better G* results for samples of 4 % to 20% rubber content by about 4.5 % to 44 % at 30 minutes and 180 °C with correlation coefficient ($R^2 = 0.98$ and 0.99) and about 6.5 % – 52 % at 60 minutes and 180 °C with correlation coefficient of ($R^2 = 0.98$ and 0.99) respectively as shown in the figures.

Tables 4.34 and 4.35 show the two-way ANOVA ($\alpha = 0.05$) of the complex shear modulus. This is evidence of a significant difference in blending temperature and crumb rubber content on G* values with P – values less than the assumed (0.0011 and 0.0010) and F value more than the F critical (of about3.8 and 3.9) respectively.

This finding indicates that the modified binder has become stiffer and less susceptible to changes in temperature. Based on these results, the complex shear modulus decreased as temperature increased from 30 °C to 80 °C; this was observed for all CRM contents under both blending time and temperature conditions, as shown in Tables TA 1– TA 23 in Appendix A.

Table 4.33: Effect of CRM Binders and Blending Time at 160 °C on G* at 76 °C

Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	6700177	15	478584.1	4.060362	0.000784	2.063541
Blending Time	30060912	2	15030456	1.520119	0.88223	3.340386
Error	3300285	28	117867.3			
Total	40061374	44				

Table 4.34: Effect of CRM Binders and Blending Time at 180 °C on G* at 76 °C

Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	5180131	15	370009.4	3.93152	0.001001	2.063541
Blending Time	36465763	2	18232882	1.732777	0.33977	3.340386
Error	2635180	28	94113.56			
Total	44281074	44				

Table 4.35: Effect of CRM Binders and Blending Temperature at 30 min on G* at 76 °C Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	6017412	15	429815.1	3.884877	0.001094	2.063541
Blending Temperature	31706770	2	15853385	13.29057	0.00001	3.340386
Error	3097865	28	110638			
Total	40822047	44				

Table 4.36: Effect of CRM Binders and Blending Temperature at 60 min on G* at 76 °C

Analysis by Two- way ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
CRM content	5741582	15	410113	3.881908	0.0011	2.063541
Blending Temperature	35281356	2	17640678	16.97712	0.00001	3.340386
Error	2958124	28	105647.3			
Total	43981062	44				

4.7.2 Analysis of Phase Angle Results at 76 °C

A phase angle (δ) represents the transition from viscous to elastic solid behaviours of bitumen binders. As mentioned in the literature review, the higher values of phase angles correspond to binders that become more viscous, as well as with lower value and more elastic. In general, this reflects a trade-off between high and low temperature performances of bitumen binders (Widyatmoko and Elliot, 2008). The test results of phase angle δ at 76 °C under different blending temperatures and blending times for various crumb rubber contents are shows in Table 4.36 and has illustrated in Figures 4.25–4.28.

Table 4.37: Results of Phase Angle at 76 °C for Different Blending Conditions

Mixing time	CRM	160 °C			180 °C		
		Mean	Std. Dev.	n	Mean	Std. Dev.	n
30	0	89.4	0.2	3	89.6	0.0	3
	4	88.4	0.2	3	84.7	0.2	3
	8	81.6	0.4	3	77.3	0.2	3
	12	73.5	0.4	3	71.5	0.5	3
	16	67.3	0.5	3	63.4	0.4	3
	20	60.5	0.4	3	60.4	0.4	3
60	0	89.4	0.3	3	89.7	0.0	3
	4	88.1	0.1	3	83.5	0.4	3
	8	79.4	0.4	3	75.5	0.4	3
	12	71.9	0.1	3	69.6	0.4	3
	16	65.3	0.3	3	61.6	1.5	3
	20	59.8	0.2	3	59.5	0.5	3

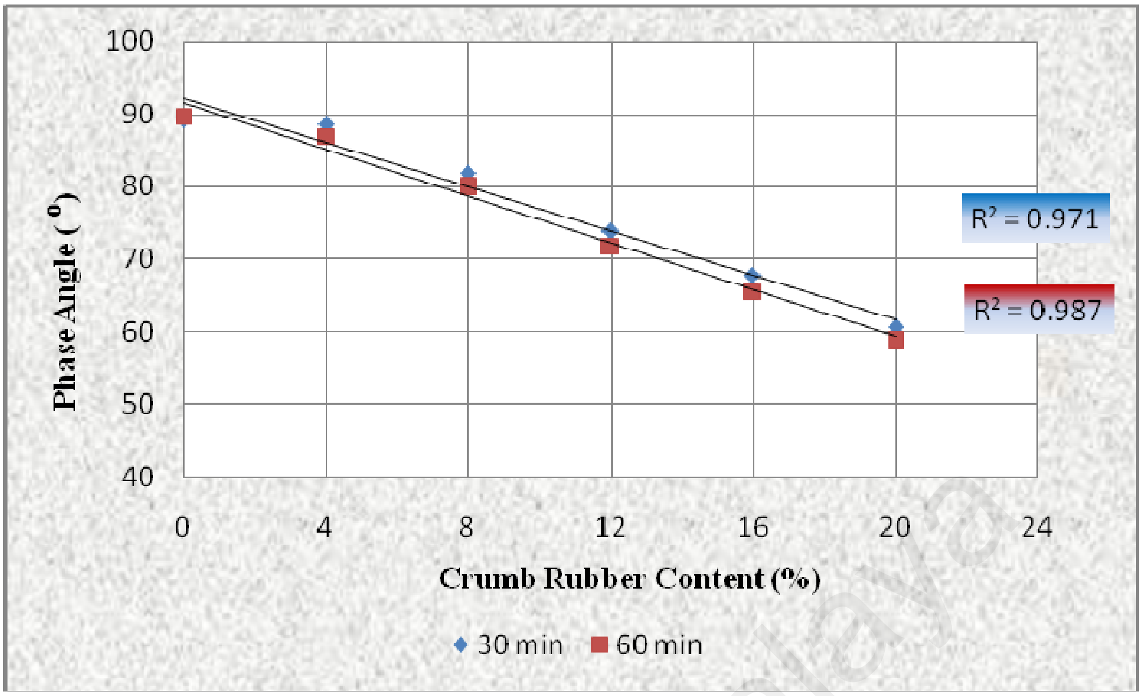


Figure 4.25: Phase Angle at 76 ^{o}C vs. Blending Time for Specimens with Blending Temperature of 160 ^{o}C

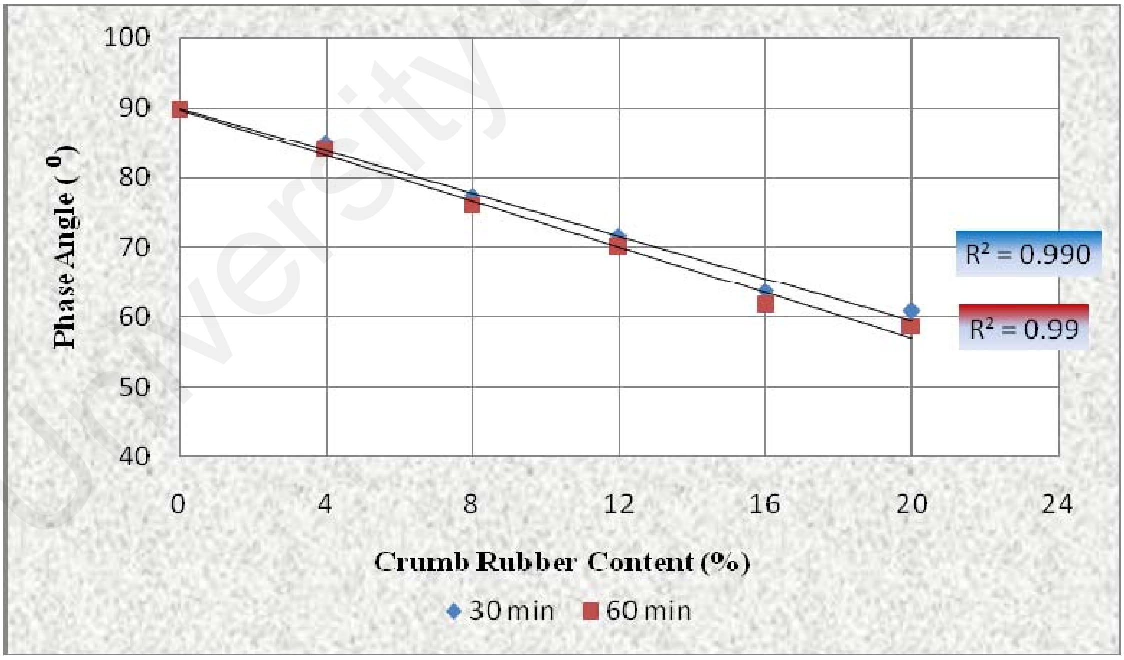


Figure 4.26: Phase Angle at 76 ^{o}C vs. Blending Time for Specimens with Blending Temperature of 180 ^{o}C

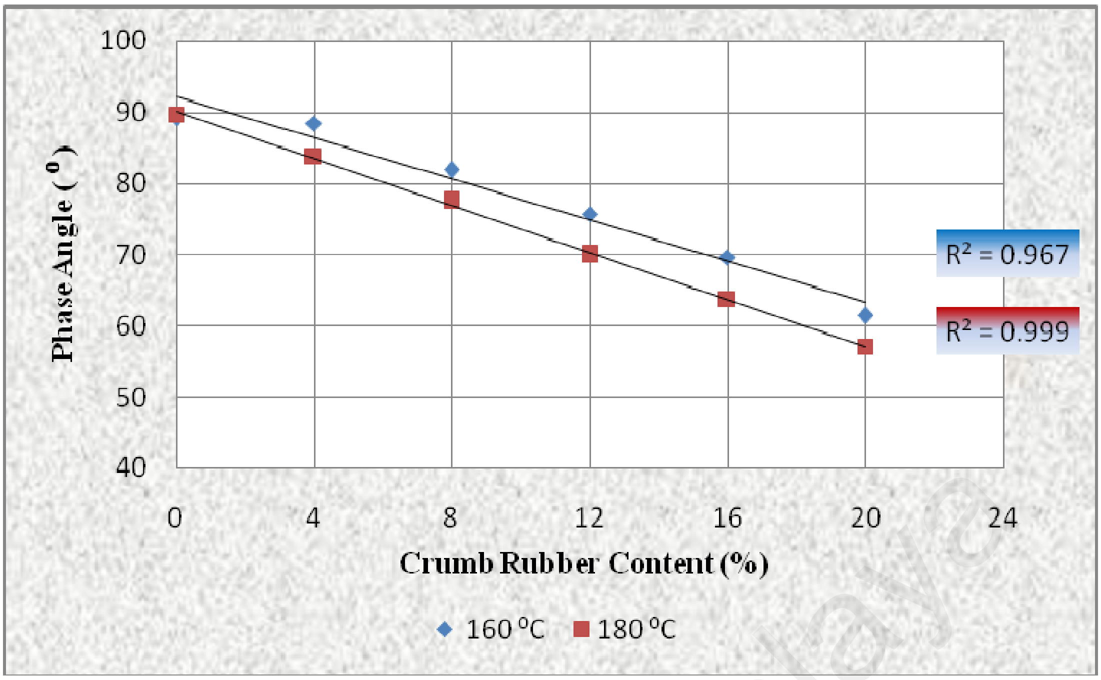


Figure 4.27: Phase Angle at 76 °C vs. Blending Temperature for Specimens with Blending Time of 30 min.

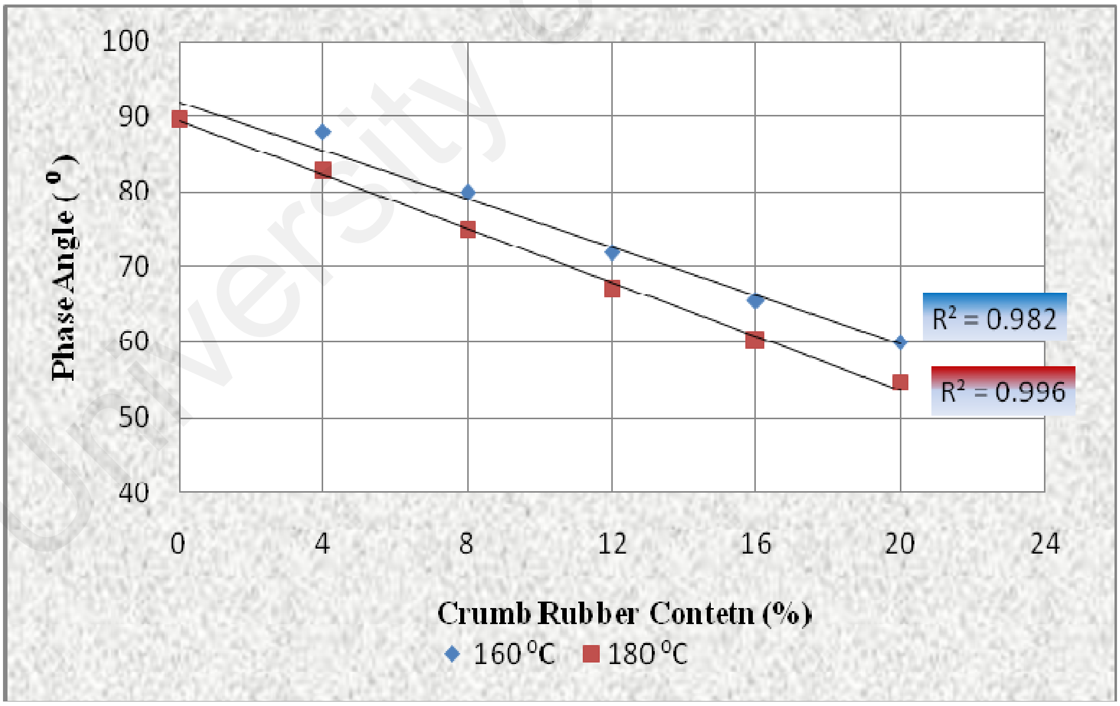


Figure 4.28: Phase Angle at 76 °C vs. Blending Temperature for Specimens with Blending Time of 60 min

4.7.2.1 Effect of Crumb Rubber Content on Phase Angle at 76 °C

Figures 4.25- 4.28 show a significant decrease in phase angle as the rubber content increases in the bituminous specimens. The decrease is linearly dependent with correlation factor $R^2 > 0.93$ as shown in the figures.

Tables 4.37 – 4.40 show the statistical analysis results the two-way ANOVA of the phase angle test (δ) at 76 °C under different blending conditions for various crumb rubber content. It is evident that the crumb rubber content has a significant effect on phase angle, as F ratio was more than F critical.

4.7.2.2 Effect of Blending Time on Phase Angle

Figures 4.25 and 4.26 show the effects of blending time on the phase angle at 76 °C. It is evident that blending time does not affect the properties of rubberised binder in terms of phase angle. Figure 4.25 shows that the decrease in phase angle for samples of 4 to 20 % rubber content, at blending temperature of 160 °C, were about 3 °C to 31 °C respectively. A similar trend was found in Figure 4.26 which shows the decrease in phase angle of modified binder samples were about 6.1 °C to 31 °C for samples of 4 to 20 % rubber content at 180 °C temperature mixes.

Tables 4.37 and 4.38 show the ANOVA ($\alpha = 0.05$) of different blending time and various crumb rubber content on the phase angle. The analysis shows evidence of insignificant different of blending time of 30 and 60 minutes on phase angle of rubberised bitumen binder. The p-values of blending time is more than the assumed value by about 0.51 and 0.67 with F- values less than F critical by about 1 and 2.1 respectively. Whereas the same analysis shows that the crumb rubber content has a significant difference on phase angle of rubberised binder as P- values are less than

(0.05) and F value are more than F critical. Thus, the primary reduction in the phase angle could be attributed to the effect of rubber content. Enhancing the rubber content led to the increase in carbon black reacting with the natural rubber, which corresponded to the elastic part of the crumb rubber chemistry.

4.7.2.3 Effect of Blending Temperature on Phase Angle at 76 °C

Figures 4.27 and 4.28 displayed the effect of blending temperature and rubber content on the phase angle at 76 °C. The blending temperature does affect the properties of rubberised binder in terms of phase angle. The increase in crumb rubber content also led to a decrease in the phase angle of rubberised binder. Figure 4.27 is shows that the decreasing phase angle for samples 4 to 20 % rubber content was about 7 °C to 35 °C, respectively. In addition, Figure 4.28 shows a decrease in phase angle as crumb rubber content increased from 4 to 20 % rubber by about 6 °C to 32 °C respectively.

Tables 4.39 and 4.40 showed the ANOVA analysis of blending temperatures and crumb rubber content effect on phase angle of rubberised binder. It is clear that both the blending temperature and crumb rubber content have a significant effect on the phase angle of the rubberised binder as P-values are less than the assumed values as well as the F values are more than the F critical. Thus, the increase in the phase angle would lead to improve in the elasticity of the modified binder and lead to better rutting resistance.

Table 4.38: Effect of CRM Binders and Blending Time at 160 °C on Phase Angle at 76 °C Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	1981.433	15	141.5309	3.914051	0.001035	2.063541
Blending Time	53956.83	2	26978.42	4.090754	0.51149	3.340386
Error	1012.472	28	36.1597			
Total	56950.74	44				

Table 4.39: Effect of CRM Binders and Blending Time at 180 °C on Phase Angle at 76 °C Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	1575.943	15	112.5674	3.90729	0.001048	2.063541
Blending Time	49848.87	2	24924.44	2.144141	0.67462	3.340386
Error	806.6682	28	28.80958			
Total	52231.49	44				

Table 4.40: Effect of CRM Binders and Blending Temperature at 30 min on Phase at 76 °C Angle Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	1758.507	15	125.6076	3.842777	0.001186	2.063541
Blending Temperature	52966.03	2	26483.02	10.20838	0.000024	3.340386
Error	915.2268	28	32.68667			
Total	55639.77	44				

Table 4.41: Effect of CRM Binders and Blending Temperature at 60 min on Phase at 76 °C Angle Analysis by Two- way ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
CRM content	1826.432	15	130.4594	3.879879	0.001104	2.063541
Blending Temperature	50769.77	2	25384.88	7.949501	0.000012	3.340386
Error	941.4891	28	33.62461			
Total	53537.69	44				

4.7.3 Analysis of Storage Modulus and Loss Modulus Results at 76 °C

During the curing process and construction of asphalt binders and aggregate mixes, one of the most important design requirements considered is the elastic deformation of bitumen binder, including its viscous flow behaviour, as the base binder (bitumen) is considered viscous to elasticity depending on loading stresses and temperatures. To understand further the various behaviours resulting from the viscous to elastic binder, the two components storage G' and loss modulus G'' were analysed. The scientific results of the main mechanism of modified and unmodified binders under different blending conditions were determined by using various crumb rubber contents.

4.7.3.1 Effect of Crumb Rubber Content on G' and G'' at 76 °C

The results of the storage modulus are shown in Figures 4.29–4.32, while the results of loss modulus are presented in Figures 4.33–4.36. Both modulus results are presented in Tables 4.41- 4.44.

Table 4.42: Viscous- elastic Properties using Blending Conditions of (160 ˚C, 30min) at 76 °C

DSR properties	Unmodified Binder 0%	Rubberised Bitumen				
		4 %	8%	12%	16%	20%
G* (pas)	235	755	1313	1872	2135	2423
δ (˚)	89.23	88.5	81.89	73.8	67.8	60.55
G' (pas)	9.6	44	140	600	721	799
G´´ (pas)	229	700	1300	1798	1977	2110

Table 4.43: Viscous- elastic Properties Using Blending Conditions of (160 ˚C, 60 min) at76ºC

DSR properties	Unmodified Binder 0%	Rubberised Bitumen				
		4 %	8%	12%	16%	20%
G* (pas)	297	889	1452	1925	2185	2467
δ (˚)	89.67	88	79.88	71.88	65.5	59.77
G’ (pas)	12.12	79	210	644	740	821
G´´ (pas)	297	888	1430	1830	1989	2132

Table 4.44: Viscous- elastic Properties Using Blending Conditions of (180 ˚C, 30min) at 76˚C

DSR properties	Unmodified Binder 0%	Rubberised Bitumen				
		4 %	8%	12%	16%	20%
G* (pas)	308	1001	1608	2011	2232	2406
δ (˚)	89.67	84.9	77.5	71.7	63.78	60.77
G’ (pas)	13.45	90	298	710	785	833
G´´ (pas)	308	998	1570	1910	2003	2199

Table 4.45: Viscous- elastic Properties Using Blending Conditions of (180 ˚C, 60min) at 76˚C

DSR properties	Unmodified Binder 0%	Rubberised Bitumen				
		4 %	8%	12%	16%	20%
G* (pas)	262	1005	1842	2069	2292	2566
δ (˚)	89.67	83.99	75.99	69.99	62.88	59.0
G’ (pas)	14.1	100	355	730	820	911
G´´ (pas)	262	1000	1788	1945	2022	2200

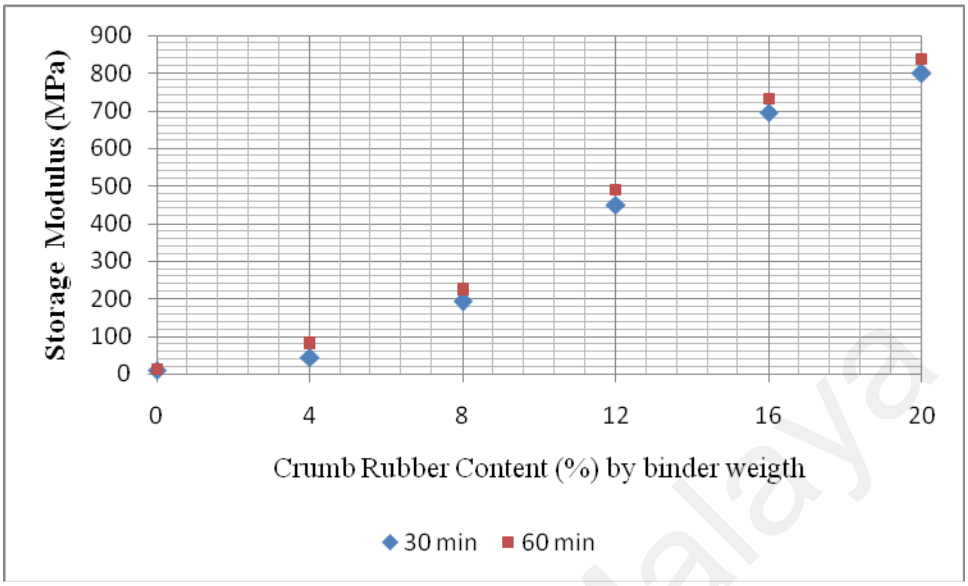


Figure 4.29: Storage Modulus at 76 ºC vs. Blending Time for Specimens with Blending Temperature of 160 ºC

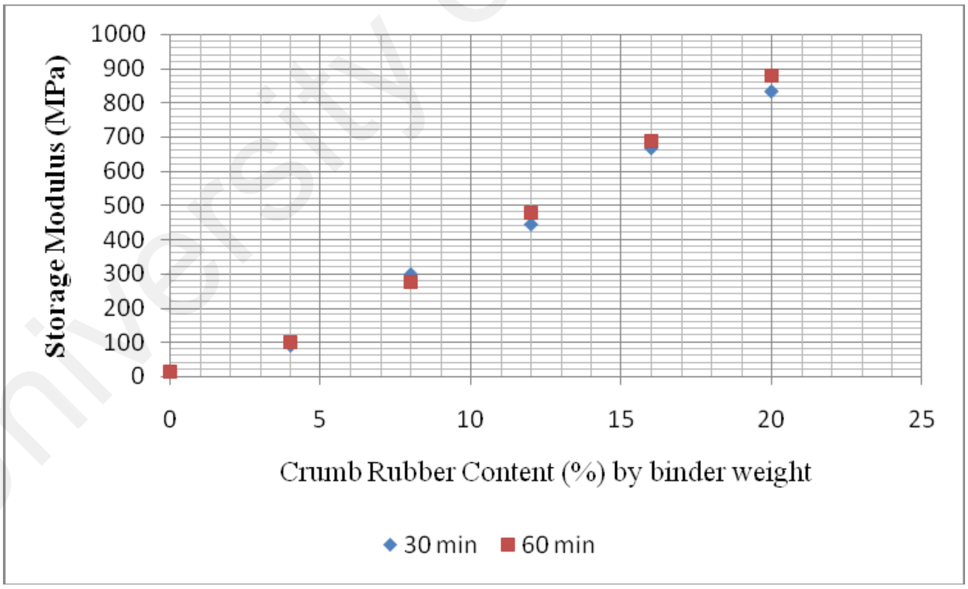


Figure 4.30: Storage Modulus at 76 ºC vs. Blending Time for Specimens with Blending Temperature of 180 ºC

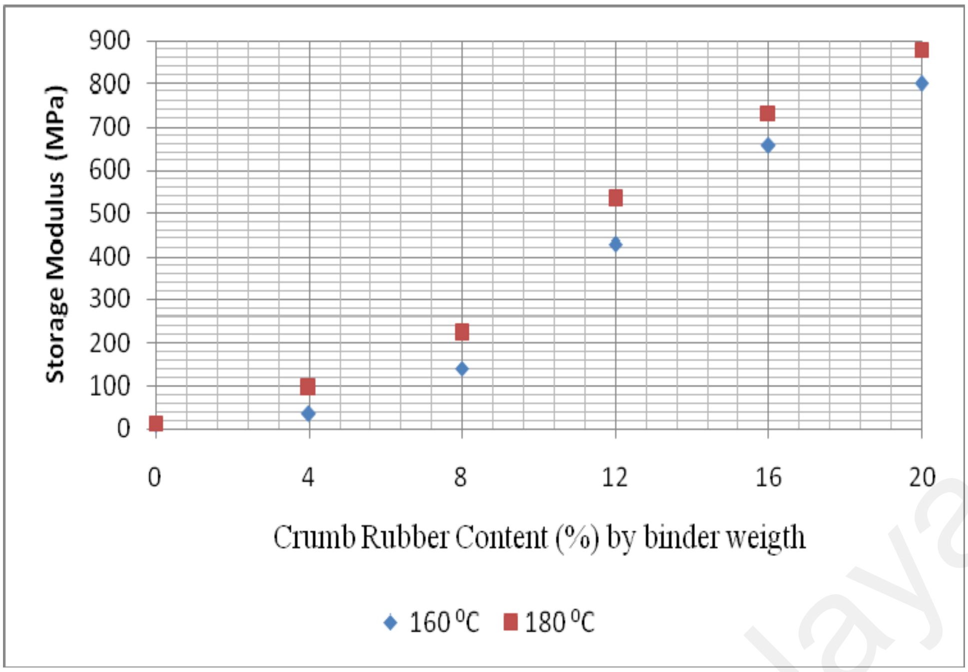


Figure 4.31: Storage Modulus at 76 ⁰C vs. Blending Temperature for Specimens with Blending Time of 30 min

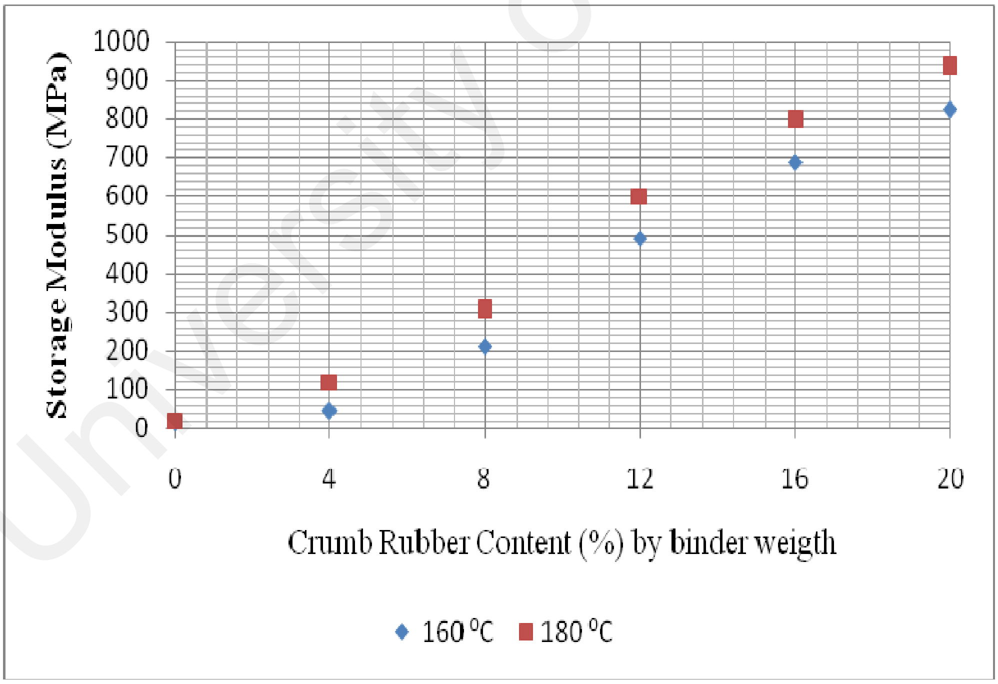


Figure 4.32: Storage Modulus at 76 ⁰C vs. Blending Temperature for Specimens with Blending Time of 60 min

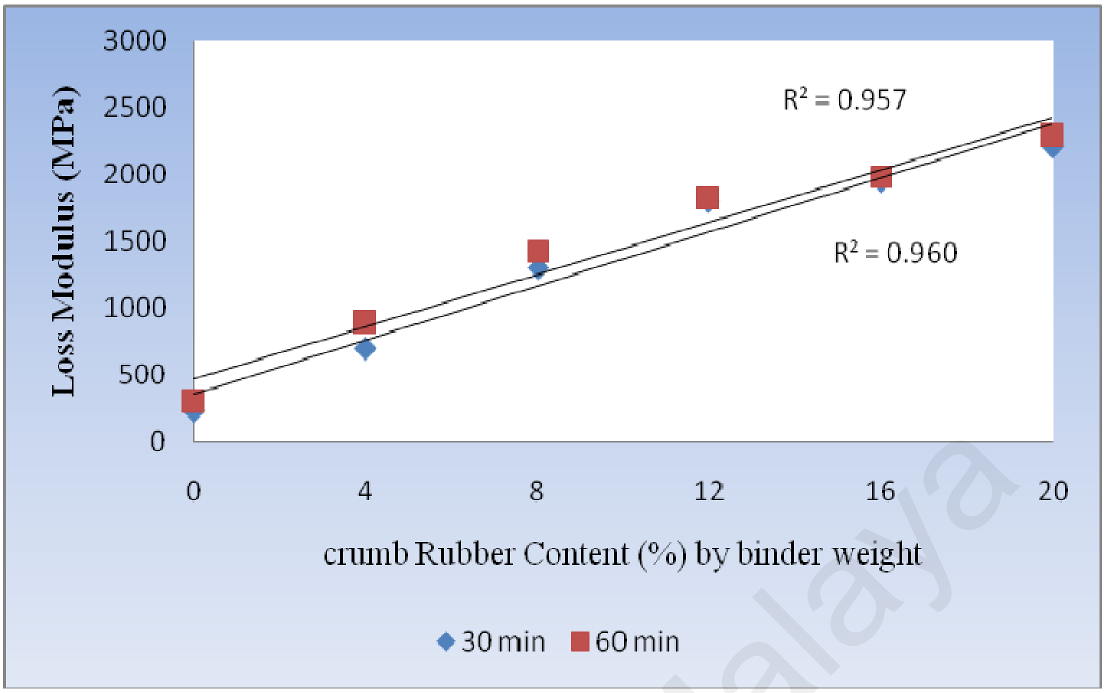


Figure 4.33: Loss Modulus at 76 °C vs. Blending Time for Specimens with

Blending Temperature of 160 °C

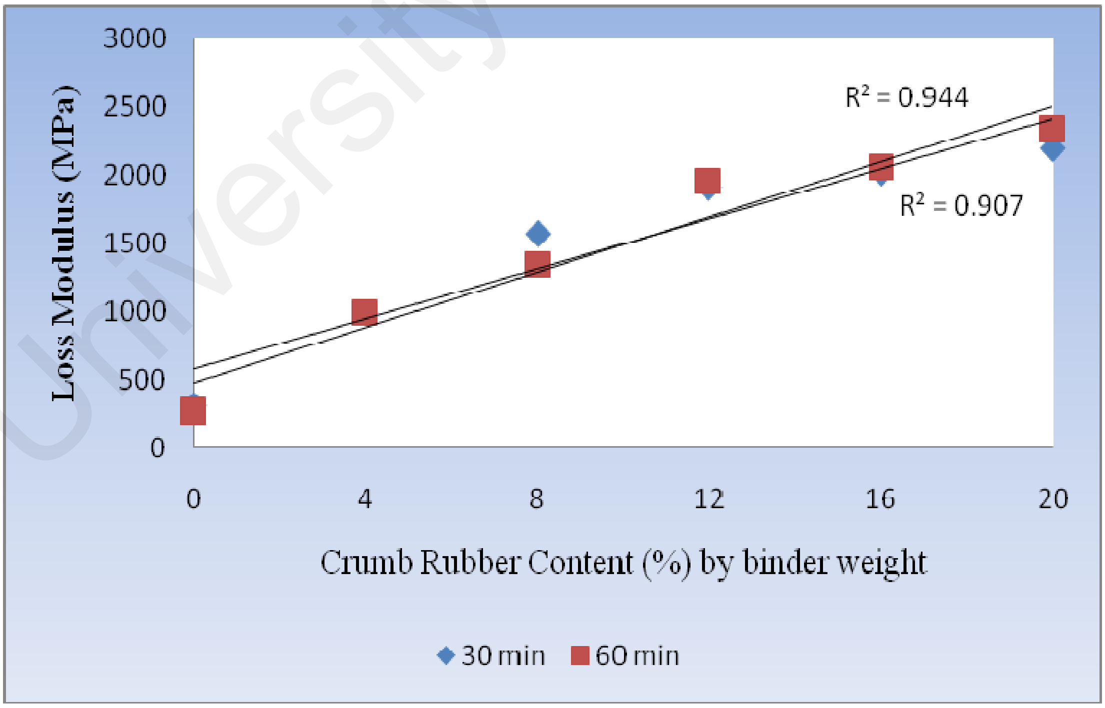


Figure 4.34: Loss Modulus at 76 °C vs. Blending Time for Specimens with

Blending Temperature of 180 °C

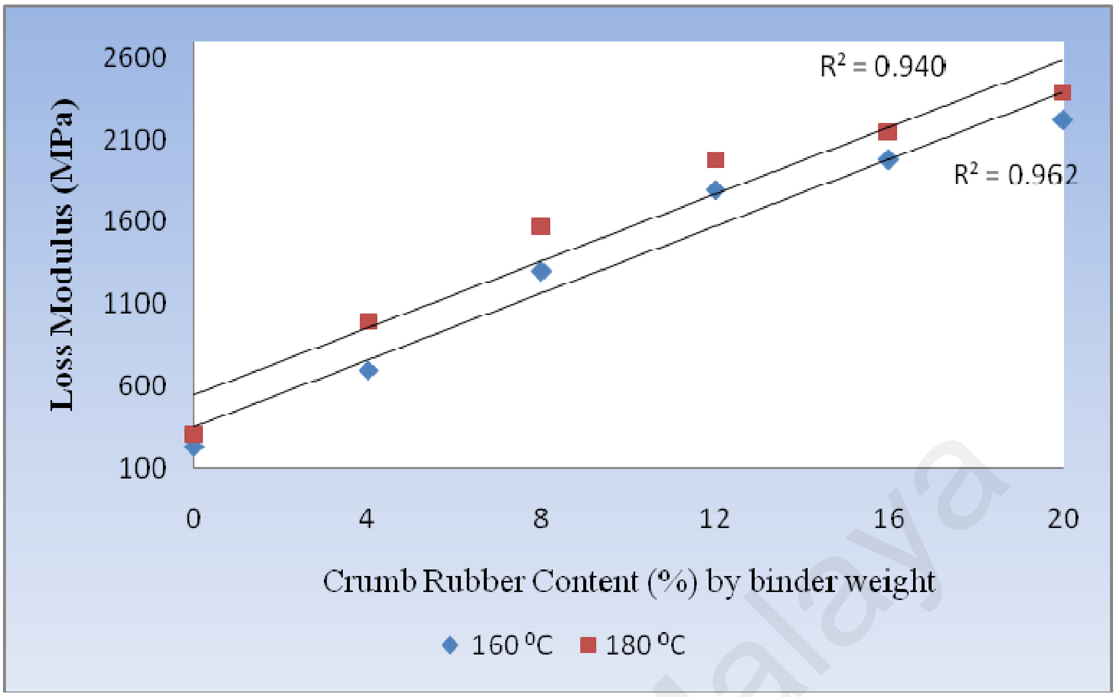


Figure 4.35: Loss Modulus at 76 °C vs. Blending Temperature for Specimens with Blending Time of 30 min

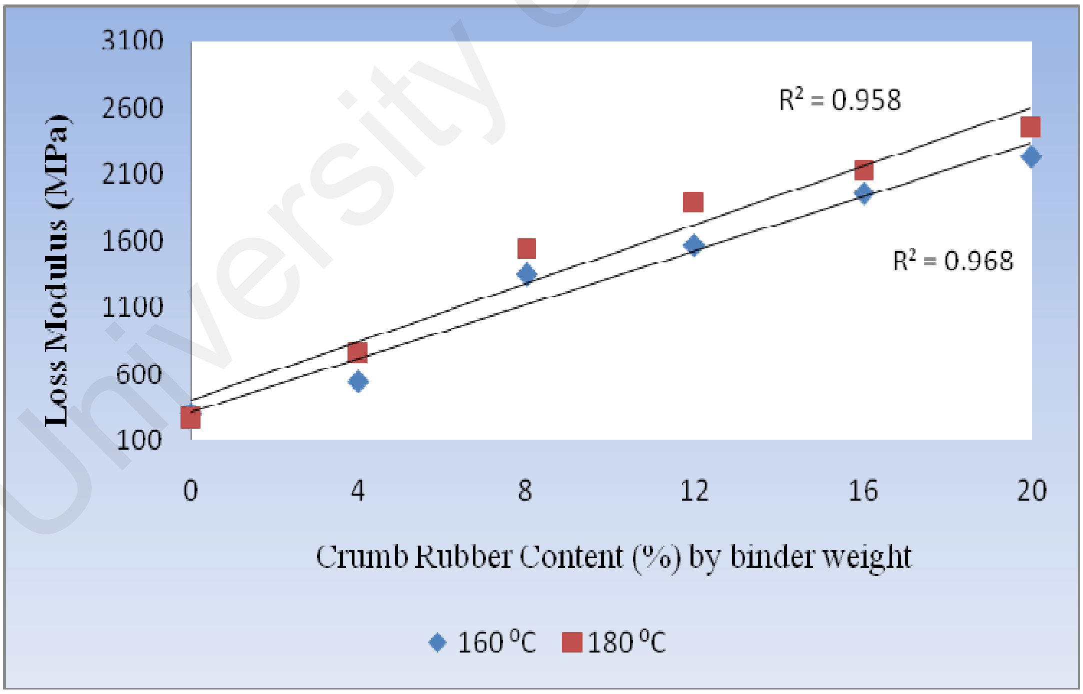


Figure 4.36: Loss Modulus at 76 °C vs. Blending Temperature for Specimens with Blending Time of 60 min

The results showed that storage modulus G’ and loss modulus G’’ appears to increased as the crumb rubber content increases. These results follow the findings of (Bouzid *et al.* 1998; Jeong *et al*., 2010). In this current study, adding crumb rubber to the base bitumen increased both storage and loss modulus over the range of blending conditions. Given that the crumb rubber might dissolve and disperse into the bitumen, the mechanical properties of the modified binder could be enhanced. Another reason would be physical chemical properties of both bitumen and crumb rubber. In general, these properties are affected during interaction processes because rubber particle dimensions are reduced, and these are related to breakdown or depolymerisation of rubber particles digested in asphalt binders (Xiao *et al*., 2006).

4.7.3.2 Effect of Blending Time on G’ and G’’ at 76 °C

The effect of blending time on storage modulus G’ showed insignificant differences between the blending times of 30 and 60 minutes for all CRM contents (see Figures 4.29 and 4.30).

Also, Figures 4.33 and 4.34 show the effect of blending time on loss modulus G’’ at 76 °C. There was minimal difference in the effect for all CRM contents under same blending conditions. This was the same with unmodified bitumen binder. In general, the higher crumb rubber content, the higher the loss modulus as shown in Figurers 4.33 and 4.34 by about 20 to 67% at 160 °C and about 28 to 79 % at 180 °C for modified samples of 4- 20% rubber content respectively.

4.7.3.3 Effect of Blending Temperature on G’ and G’’ at 76 °C

As shown in Figures 4.31 and 4.32, blending temperatures significantly affected the storage modulus G’ of rubberised bitumen binder, while same conditions of temperature showed insignificant effect on the storage modulus G' of unmodified binder. By using the respective 4% to 20% rubber contents, storage modulus G' increased from 44– 90 to 799–833 Pas with blending time of 30 min and from 97–100 to 821–911Pas with blending time of 60 min (see Figures 4.31 and 4.32). The increases indicate that blending temperature was the control factor of the blending conditions for the rubberised bitumen binders. Furthermore, these results of the storage modulus, which may enhanced the elastic response of the modified binders. The elasticity and recovery of the modified binders improved after they were subjected to shear stress and strain deformation.

Figures 4.35 and 4.36 show the effect of blending temperatures and crumb rubber content at 76 °C on loss modulus G''. A significant effect of blending temperature on loss modulus G'' is illustrated for all modified bitumen binder, while it showing no effect on unmodified bitumen binder. Higher blending temperature and increased in crumb rubber content has led to increased loss modulus by about 22- 68 Pas at 30 min and 19- 84 Pas at 60 min for samples of 4- 20 % rubber content as shown in Figures 4.35 and 4.36 respectively.

The increase in storage modulus and loss modulus could have resulted from the oxidation processes of bitumen fractions. The interaction processes of blending temperature and bending time could have dominated the visco-elastic behaviour of the rubberised bitumen binder in the temperature sweep range of 30–80 °C as shown in Tables TA 1 – TA 23 in Appendix A. The bitumen binder becomes softer at 76 °C. As these interaction condition processes might be sufficient, the binder might show predominant elastic properties. Storage modulus (elastic) was higher than loss (viscous) modulus; this phenomenon was proven by the increase in phase angle values. An ideal binder is sufficiently stiff at high temperatures in order to withstand permanent deformation (rutting) and sufficiently soft at low temperatures in order to avoid excessive thermal stresses leading to fatigue.

Tables 4.41–4.44 show the effect of different blending times, blending temperatures, and various CRM contents on loss modulus G’, storage modulus G”, complex modulus G* and phase angle δ at 76 °C. The significant increases in loss modulus G’, storage modulus G” and complex modulus G* were clearly affected by higher crumb rubber contents. Rubber mass increased during the swelling process of blend interaction at 60 min blending mix time and 180 °C blending temperature; these were similar to the findings of Jeong *et al.* (2010), who reported that longer blending time and temperature on the visco-elasticity of crumb rubbers would lead to sufficient softening of the asphalt binder.

The results of this study indicate that the crumb rubber-modified binder manifest visco-elastic behaviours, with the binder exhibiting better relaxation upon applied stress on to the asphalt rubber. In summary, the results have confirmed that rubber-modified binders could become less susceptible to temperature changes as rubber content is increased. The results further confirm improvements in asphalt performance properties at high service temperatures.

4.7.4 Analysis of Rutting Factor (G*/sin δ) Results at 76 ^{o}C

Rutting factor G*/sin δ (superpave parameter) was set as the stiffness indicator for measuring and evaluating the rutting resistance of both unmodified and polymer-modified binders (Bahia and Anderson, 1995a). SHRP has defined the requirements of rutting factor G*/sin δ, which correspond to the high-temperature viscous component of binder stiffness. Accordingly, G*/sin δ must be at least 1 KPa for unaged binders and a minimum of 2.2 KPa for aged binders in the rolling thin film oven test (RTFOT).

The rutting factor G*/sin (δ) at 76 °C under different blending times and blending temperatures for various CRM contents are shown in Table 4.45 and illustrated in Figures 4.37 –4.40.

Table 4.46: Results of G*/sin δ of CRM Binders at 76˚C for Different Blending Conditions

CRM (%)	**Rutting Factor (G*/sin δ) (Pas)**			
	B.C.160 °C,30 min	B.C.160 °C,60 min	B.M.180°C,30 min	B.C.180°C, 60 min
0	235	297	308	262
4	755	900	1004	1010
8	1326	1474	1647	1898
12	1949	2025	2118	2201
16	2305	2401	2488	2598
20	2782	2857	2972	2993

B.C.: Blending conditions.

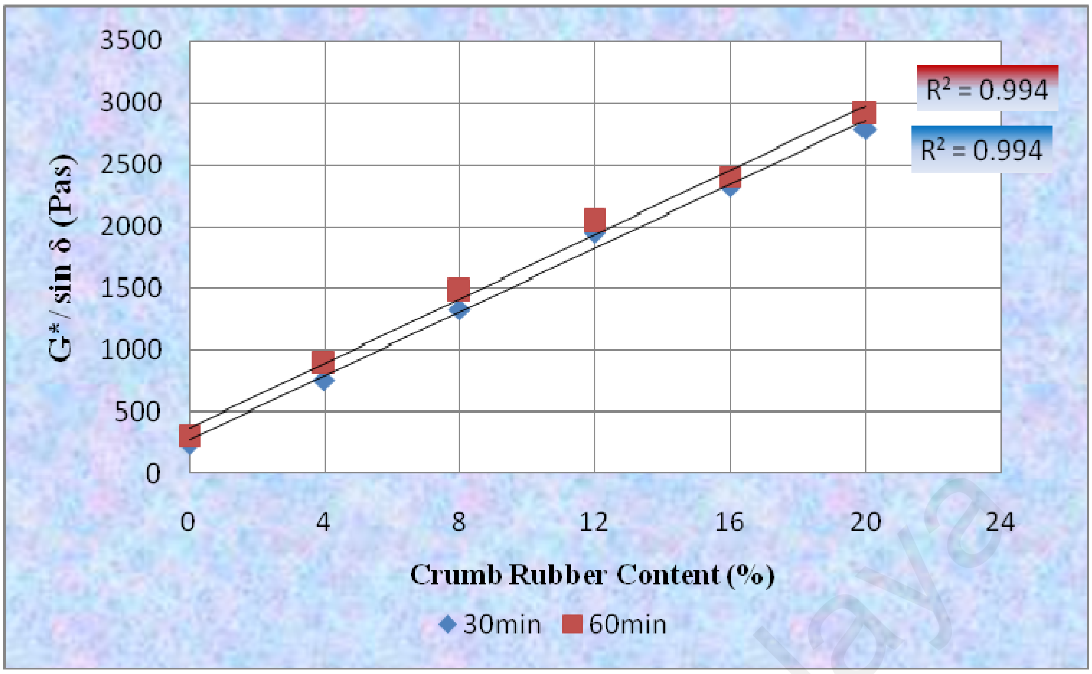


Figure 4.37: Rutting Factor at 76 °C vs. Blending Time for Specimens with Blending Temperature of 160 °C

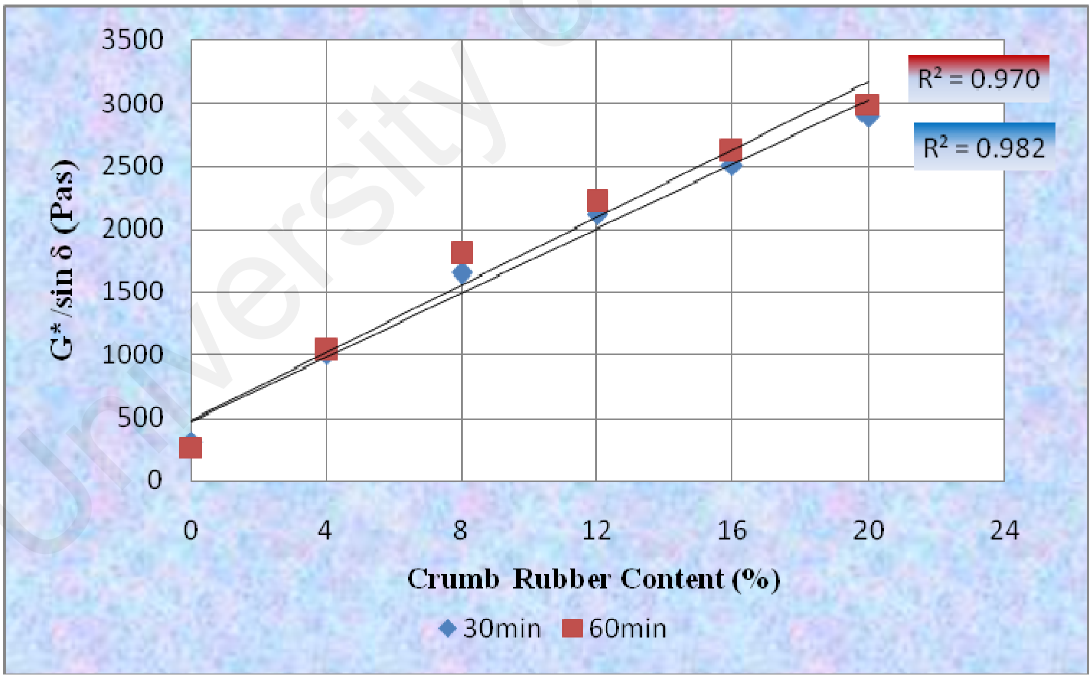


Figure 4.38: Rutting Factor at 76 °C vs. Blending Time for Specimens with Blending Temperature of 180 °C

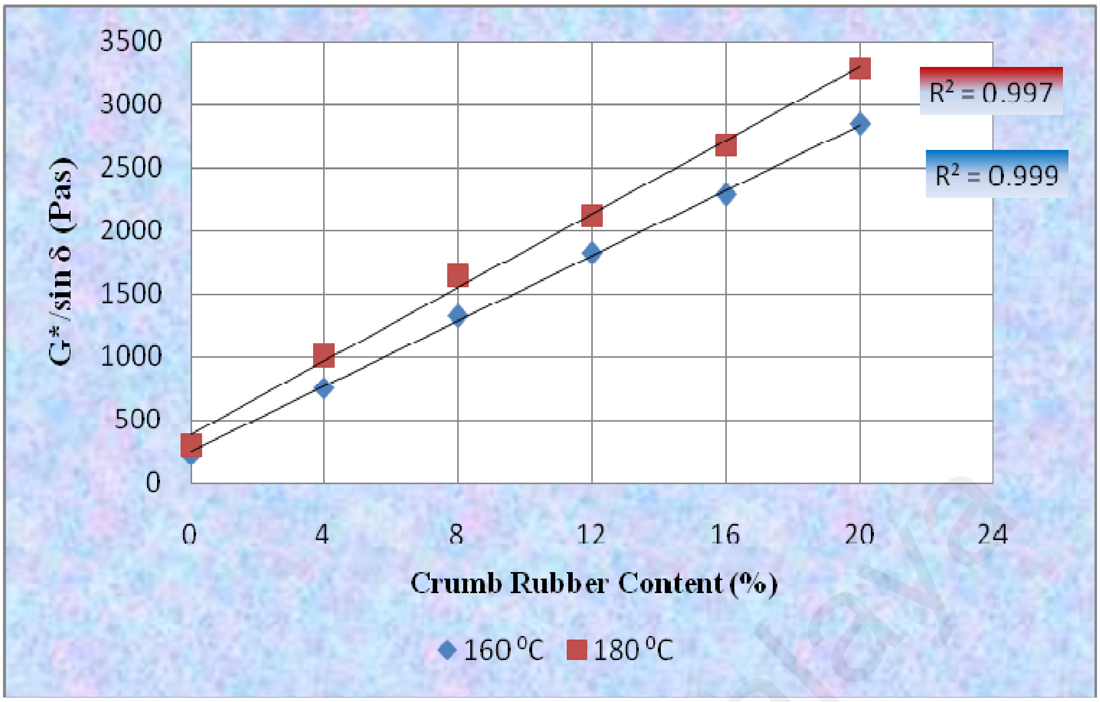


Figure 4.39: Rutting Factor at 76 °C vs. Blending Temperature for Specimens with Blending Time of 30 min

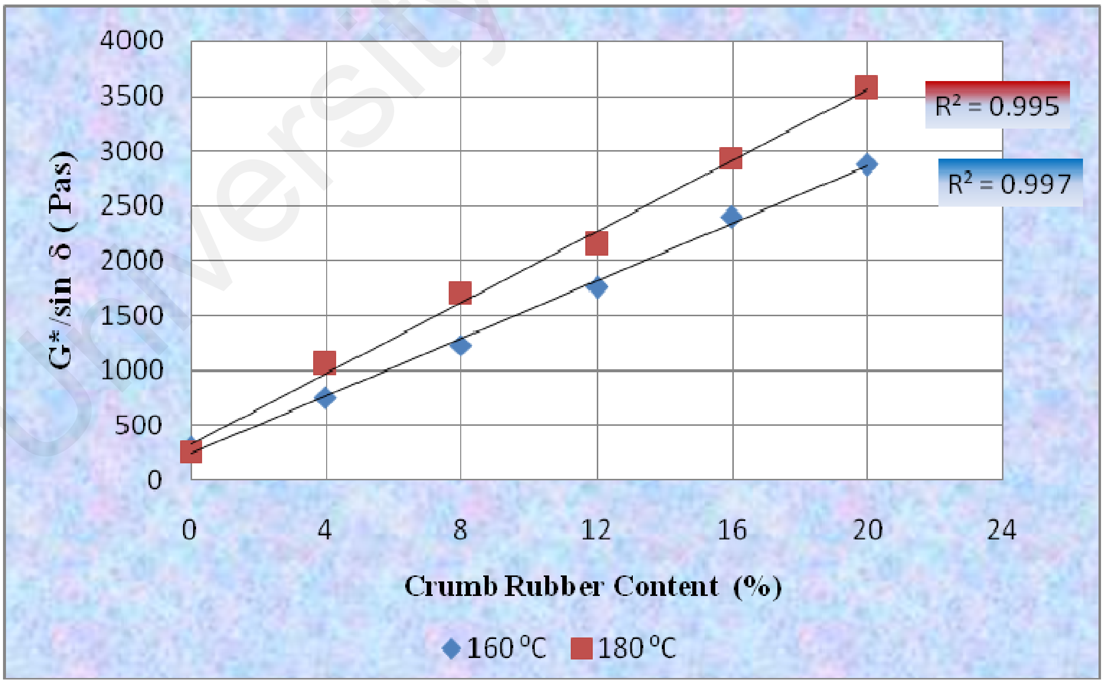


Figure 4.40: Rutting Factor at 76 °C vs. Blending Temperature for Specimens with Blending Time of 60 min

4.7.4.1 Effect of Crumb Rubber Content on G*/sin (δ) at 76 °C

Figures 4.37–4.40 showed an increase in rutting factor G*/sin (δ) at 76 °C as CRM content increased in the bitumen specimens. The increase is linearly dependent with correlation factor R^2 = 0.970, 0.982, 0.994, 0.994and 0.999 for all the figures.

The increase in crumb rubber content of samples from 4 – 20 % led to an increase in the rutting factor by about 22- 92 % respectively. Thus, the higher crumb rubber content is the main factor that lead to higher (G*/ sin δ). This is in concurrence with Jeong *et al.* (2010) who reported similar findings.

The results agree the finding of previous studies, which reported adding crumb rubber to bitumen binder would lead to the increase in G*/sin δ values (Yilidriim, 2007; Palit *et al.*, 2004; Jeong *et al.*, 2010). Adding crumb rubber increased stiffness, thus yielding a higher failure temperature. During blending, the chemical reaction between rubber and bitumen binder saw to changes in binder properties. As expected the rubber particles tend to swell due to the absorption of aromatic oils, leading to the production of viscous gels (Kim *et al.*, 2001).

4.7.4.2 Effect of Blending Time on G*/sin (δ) at 76 °C

Figures 4.37 and 4.38 show the results of rutting factor (G*/sin δ) at 76°C for the two blending time and various crumb rubber content. The difference in blending time does not appear to have any effect on the rutting factor (G*/sin δ) at 76°C, at least between the case of 30 and 60 minutes. In addition, the blending time of 30 min might not be enough to allow complete reaction between the crumb rubber and asphalt binder (Paulo and Jorge, 2008). Longer blending time, however, produced better results on performance properties of modified binder according to findings from studies by Jeong *et al.* (2010). The amount of crumb rubber in the rubberised asphalt binder, nevertheless, does significantly increase the rutting resistance of the binder. Studies by Bahia *et al.* (1998) also showed that binder resistance to rutting increases with such binder modification.

4.7.4.3 Effect of Blending Temperature on G*/sin (δ) at 76 °C

Figures 4.39 and 4.40 show the effects of blending temperatures and crumb rubber content on the rutting factor (G*/ sin δ). A significant difference **on rutting factor values a**mong all modified bitumen binders was observed except for the low rubber content 4%, which was inconstant and less than 1000 Pa (less than SHRP limits) at about 755 and 750 Pa for blending times of 30 and 60 min, respectively, at 160 °C blending temperature.

Higher blending temperature for the samples with 8%– 20% CRM contents produced better G*/sin δ results. All values were greater than the SHRP requirement of 1 KPa. These results were similar to a study made on #40 crumb rubber , which showed that longer blending time and higher blending temperature result in higher resistance to permanent deformation rutting (i.e., G*/sin δ values improves modified rubber binders) (Joeng *et al.*, 2010).

Thus, the modified binder in this study produced at 180 °C was expectedly more resistant to rutting; this was observed for all binders with 8%, 12%, 16%, and 20% contents. Based on the report on bitumen binder rutting, shear flow deformation is the main deformation mode of asphalt binders. According to SHRP guidelines, the performance grade should be based on the ability of bitumen binders to resist permanent deformation (Asphalt Institute, 2003). Thus, the samples of rubberised bitumen used in this research with rubber content of 8%–20% could improve field resistance properties during high-temperature deformation.

4.8 Effect of RTFOT and PAV Aging on Rubberised Binder

The effect of both short-term and long-term aging on the rheological properties of crumb rubber-modified bitumen binders was studied. During RTFOT, Superpave was used to measure the short-term oxidation occurring in bitumen binders through the hot mix process. Meanwhile, oven aging via the pressure aging vessel (PAV) was used to measure the effect of long-term oxidation on rubberised bitumen binders, which also corresponded to durability of modified binders in pavements.

The effect of aging and crumb rubber content on the viscous-elastic properties was also investigated. The concentrations of crumb rubbers by binder weight were 0%, 8% and 16%. DSR, penetration and ring and ball softening point for the conventional binder test were used to characterise the rheology and stiffness of binders before and after oven aging. Brookfield viscosity was applied to evaluate the linear viscosity behaviour of unmodified and modified bitumen before and after aging.

4.8.1 Effect of Rubber Content and Aging Conditions on Complex Shear Modulus G* at 76 ^{o}C

Table 4.46 shows the effect of aging conditions and crumb rubber content on **G* values** of modified and unmodified rubber bitumen binders at 76 °C. Figures 4.41 and 4.42 show the effect of crumb rubber content and aging phases/conditions on G* at76 °C respectively. As rubber content increased from 8 to 16%, complex shear modulus (G*) value showed a dramatic increase by about 1.5 to 2 times for aged binder compared to unaged binder. There was obvious difference in aging conditions at 16%

rubber content by about 2491.7 to 3445.3 of PAV and RTFOT respectively as illustrated in Figure 4.42. In addition, the modified binder was also of a higher G* value than the unmodified by about 1.5–2.5 times for samples with 8 and 16% rubber content. Thus, the increase in crumb rubber content had an obvious effect on aged rubberised bitumen rutting factor compared to aged unmodified bitumen with correlation coefficient R^2 = 0.928, 0. 891 and 0.995 for unaged, RTFOT and PAV aging conditions respectively. These results are in tandem with the findings of (Mahrez and Rehan, 2003).

Oxidative aging might have increased or decreased the temperature susceptibility of the stiffness of the modified binder, which could have led to the increase in asphaltene. Ruan *et al*. (2003) stated that the decrease in temperature susceptibility of the modified binder showed better durability in terms of high-temperature lifetime service of pavements. Essentially, the increase in shear modulus led to oxidative degradation of rubber, as well as to low molecules that could harden the base binder. Oxidative aging change could also affect the base bitumen binder and the rubber polymer degradation, which could lead to diminished aging; in effect, the binder would become more stiff and durable, with long-term maintenance and greater resistance to rut deformations.

In general, the results of aged rubberised binder were in concurrence with the Superpave binder specification of a minimum G* /sin δ requirment using RTFOT (2.2 Pa) , thus the higher G* /sin δ values indicated that the binders would be less susceptible to rutting defromation at high pavemant temperatures (The Asphalt Institute, 2003).

Figure 4.43 shows the aging index of rutting factor, G*/ sin δ, of original and crumb rubber modified bitumen binder at 76 °C as calculated by Eqn. 4.1 as:

$$AIRF = (G^*/\sin\delta)_{aged} / (G^*/\sin\delta)_{unaged} \quad \text{Eqn. 4.1}$$

Where AIFR is the aging index of rutting factor, (G*/sin δ) aged and (G*/sin δ) unaged are the G*/sin δ values of bitumen binder after and before aging respectively.

The results exhibited that the G* /sin δ of bitumen binder containing different content of crumb rubber increased after two different aging, especially after PAV aging. But, the slope of the G* /sin δ decreased with increased crumb rubber concentration, meaning that crumb rubber made the bitumen binder resist thermo-oxidation aging.

Table 4.47: Results of Complex Shear Modulus G*of Aged Rubberised Bitumen at 76 °C

Complex shear modulus G* (MPa)				
Group	CRM	Mean	Std. Deviation	*n
Control (unaged)	0%	261.3	3.06	3
	8%	1842.7	12.22	3
	16%	2293.7	5.69	3
RTFOT	0%	502.0	11.53	3
	8%	2316.3	6.51	3
	16%	2491.7	4.04	3
PAV	0%	921.0	8.19	3
	8%	2459.3	4.51	3
	16%	3445.3	6.51	3

*n: number of samples

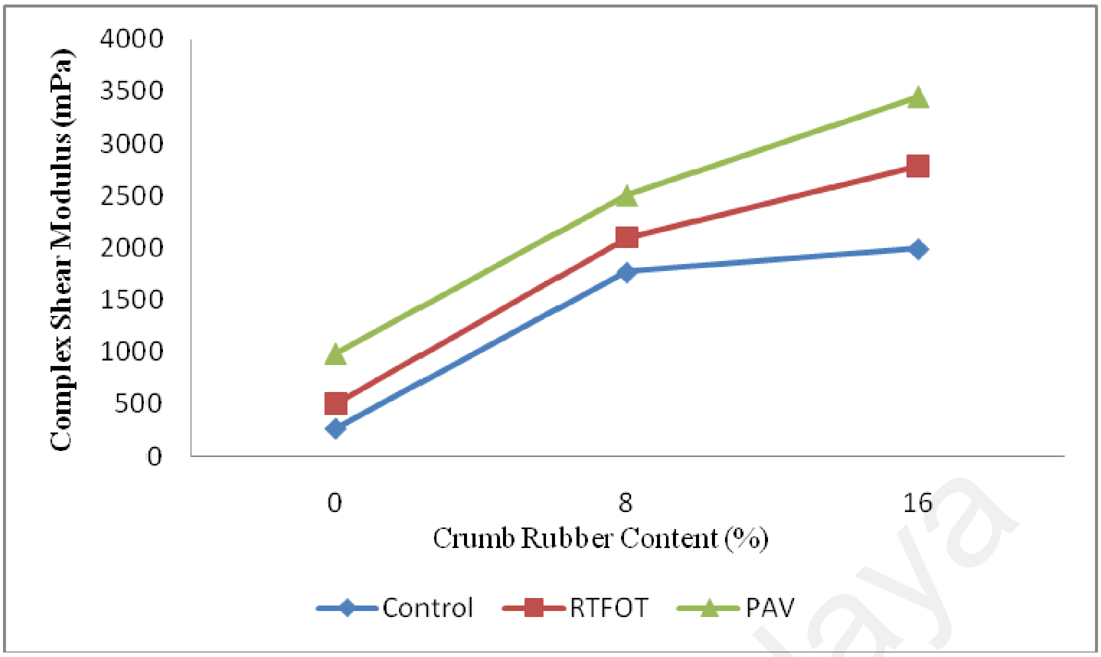


Figure 4.41: Complex Shear Modulus vs. Crumb Rubber Content at 76˚C

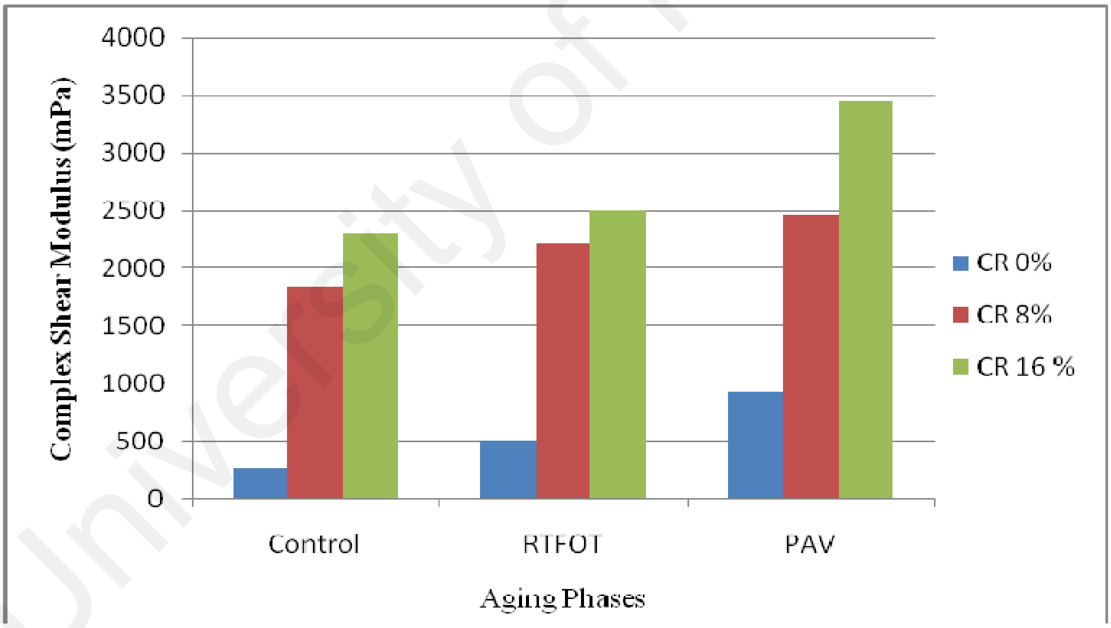


Figure 4.42: Complex Shear Modulus vs. Aging Phases of Specimens at 76 °C

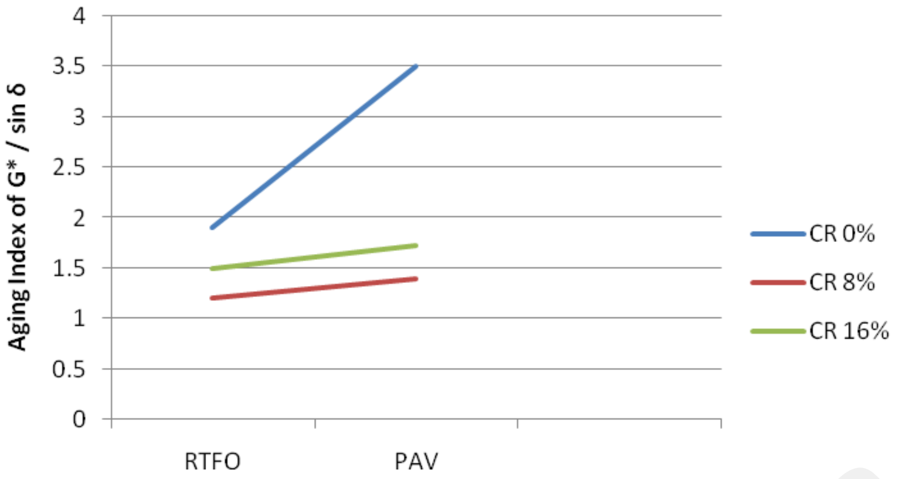


Figure 4.43: Aging index of G*/sin δ of bitumen binder with different CRM content

4.8.2 Effect of Aging Conditions and Rubber Content on Phase Angle at 76 °C

Phase angle was used to measure and determine elastic stability under high temperatures. Findings showed the visco-elastic response of the bitumen binder. As shown in Table 4.47, phase angle δ for the unmodified bitumen is higher than rubberised bitumen. In general, as crumb rubber content increased, phase angle δ decreased for the various aging methods. Increases in crumb rubber percentage led to varying degrees in phase angles, indicating increase in elastic behaviour of the binder.

Figure 4.44 and 4.45 show the effect of crumb rubber content on δ at76 °C respectively. As rubber content increased from 8 to 16%, δ value showed dramatic decrease by about 1.0 to 1.5 times for aged binder compared to unaged binder.

Since phase angle is a measure of the ratio between loss modulus G' and loss modulus G" (tan δ = G"/ G'), the degrees of phase angle δ implies that aging led to greater increase in storage modulus compared with those in loss modulus.

Table 4.48: Results of Phase Angle of Aged Rubberised Bitumen at 76˚C

Phase angle δ (°)				
Group	CRM	Mean	Std. Deviation	n
Control (unaged)	0%	89.8	0.12	3
	8%	76.5	0.45	3
	16%	62.8	0.12	3
RTFOT	0%	85.0	0.40	3
	8%	75.4	0.46	3
	16%	64.1	0.34	3
PAV	0%	86.4	0.50	3
	8%	72.8	0.23	3
	16%	61.4	0.50	3

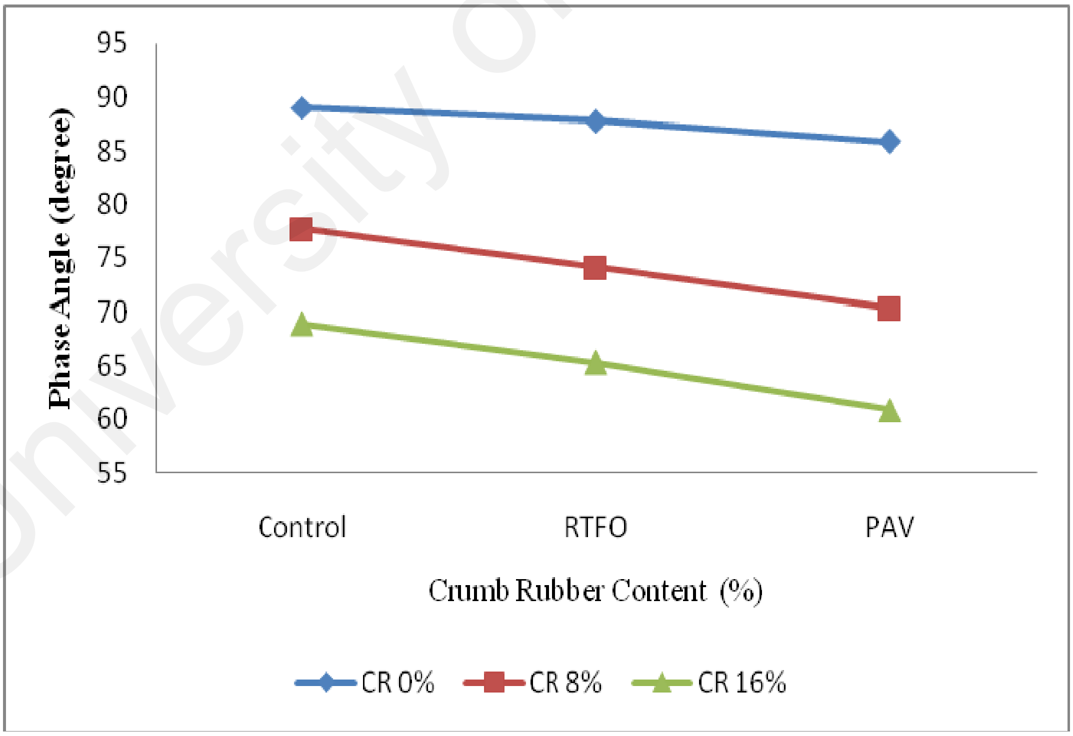


Figure 4.44: Phase Angle vs. Crumb Rubber Content at 76 °C

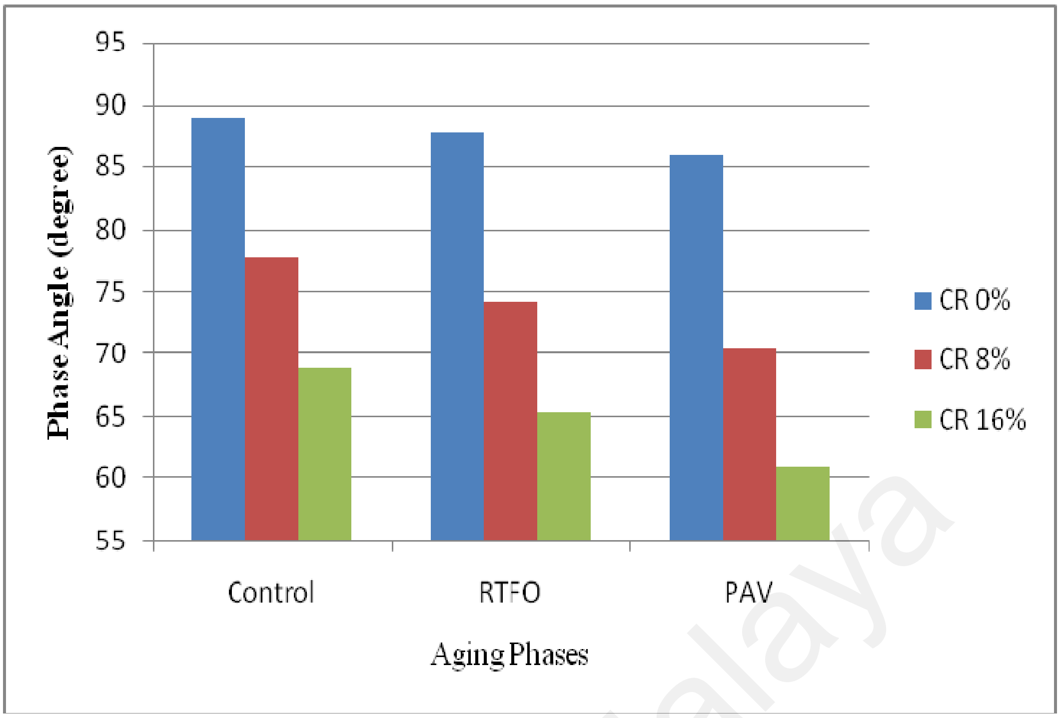


Figure 4.45: Phase Angle vs. Aging Phases of Specimens at 76 °C

4.8.3 Analysis of Storage Shear Modulus and Loss Shear Modulus after Aging at 76 °C

Storage modulus G’ can be defined as the main part of the complex shear modulus and is associated with storage energy during recoverable deformation. Loss modulus G’’ can be defined as the imaginary part of complex shear modulus associated with the dissipation of energy through permanent deformation. In general, for a typical visco-elastic material like bitumen, the storage modulus is higher than loss modulus.

Figures 4.46, 4.47, 4.48, 4.49 and Tables 4.48 and 4.49 show the effect of various crumb rubber contents for the storage shear modulus and loss shear modulus respectively. Under different rubber content, storage modulus was increased in the rubberised bitumen samples. Whereas loss modulus exhibited only a slight effect on rubberised bitumen samples (see Figures 4.46, 4.47, 4.48 and 4.49).

However, although both modulis increased significantly, storage modulus has higher value compared with loss modulus. This was confirmed by the decrease in phase angle, indicating a more elastic behaviour. As rubber content increased, the oxidation aging of modified binders increased the asphaltense in the base asphalt and decreased the molecular size of rubber modifiers.

Table 4.49: Results of Storage Shear Modulus of Aged Rubberised Bitumen at 76˚C

Storage Shear Modulus (MPa)				
Group	CRM	Mean	Std. Deviation	n
Control (unaged)	0%	7.5	0.45	3
	8%	354.0	17.35	3
	16%	780.7	7.51	3
RTFOT	0%	43.7	6.51	3
	8%	412.7	7.02	3
	16%	981.0	7.55	3
PAV	0%	49.3	9.02	3
	8%	487.7	7.51	3
	16%	1219.7	5.51	3

Table 4.50: Results of Loss Modulus of Aged Rubberised Bitumen 76˚C

Loss Modulus (MPa)				
Group	CRM	Mean	Std. Deviation	n
Control (unaged)	0%	260.0	2.00	3
	8%	1788.0	5.00	3
	16%	2021.7	3.51	3
RTFOT	0%	496.3	4.73	3
	8%	2208.7	8.50	3
	16%	2310.7	4.04	3
PAV	0%	621.0	4.58	3
	8%	2411.7	8.02	3
	16%	2987.0	7.00	3

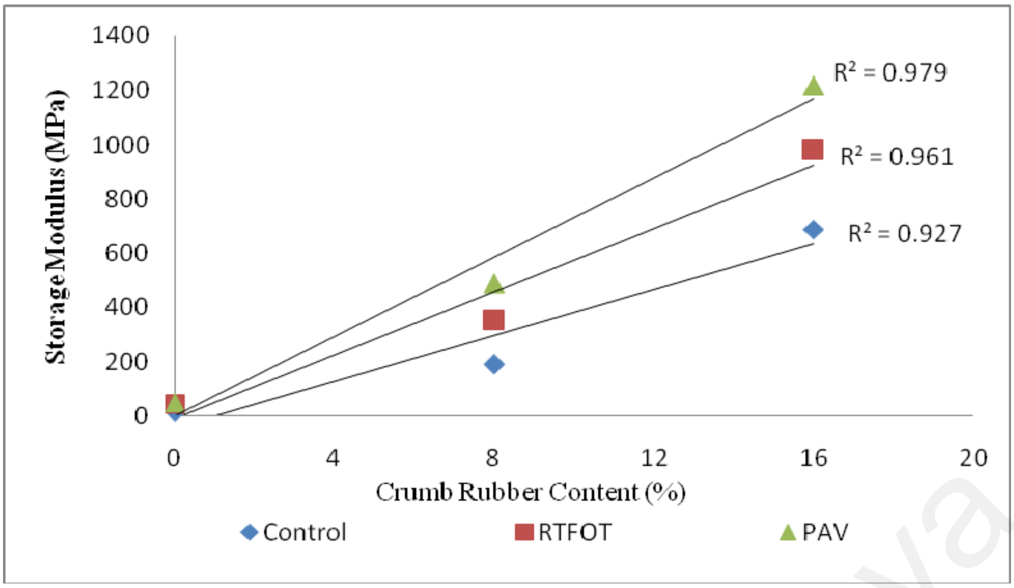


Figure 4.46: Storage Shear Modulus vs. Crumb Rubber Content of samples at 76˚C

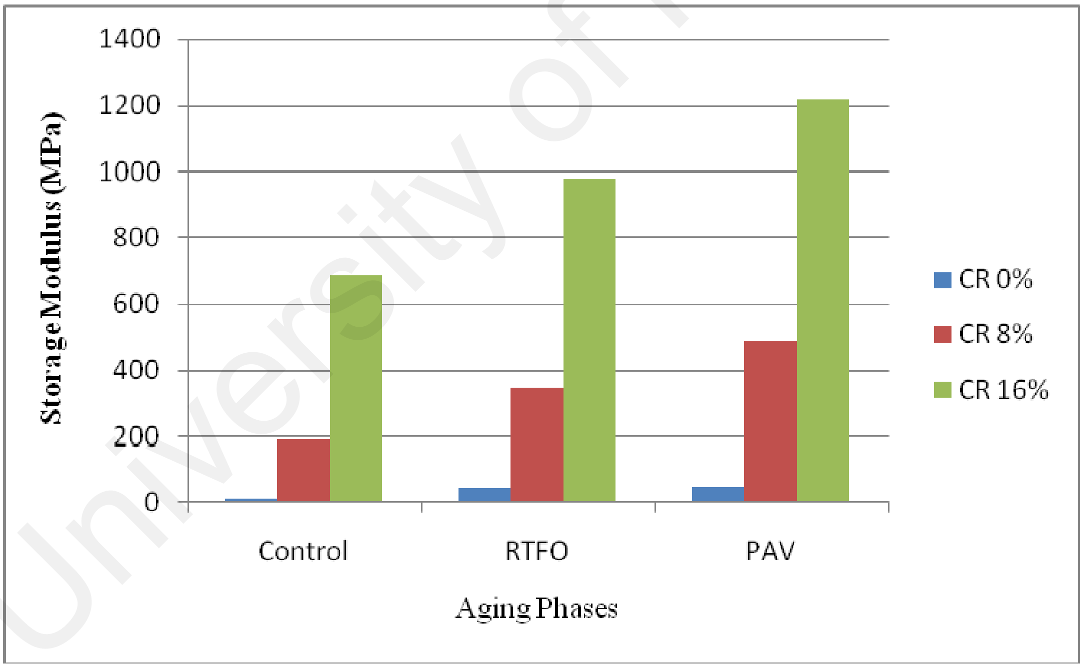


Figure 4.47: Storage Shear Modulus vs. Aging Phases for samples at 76 ˚C

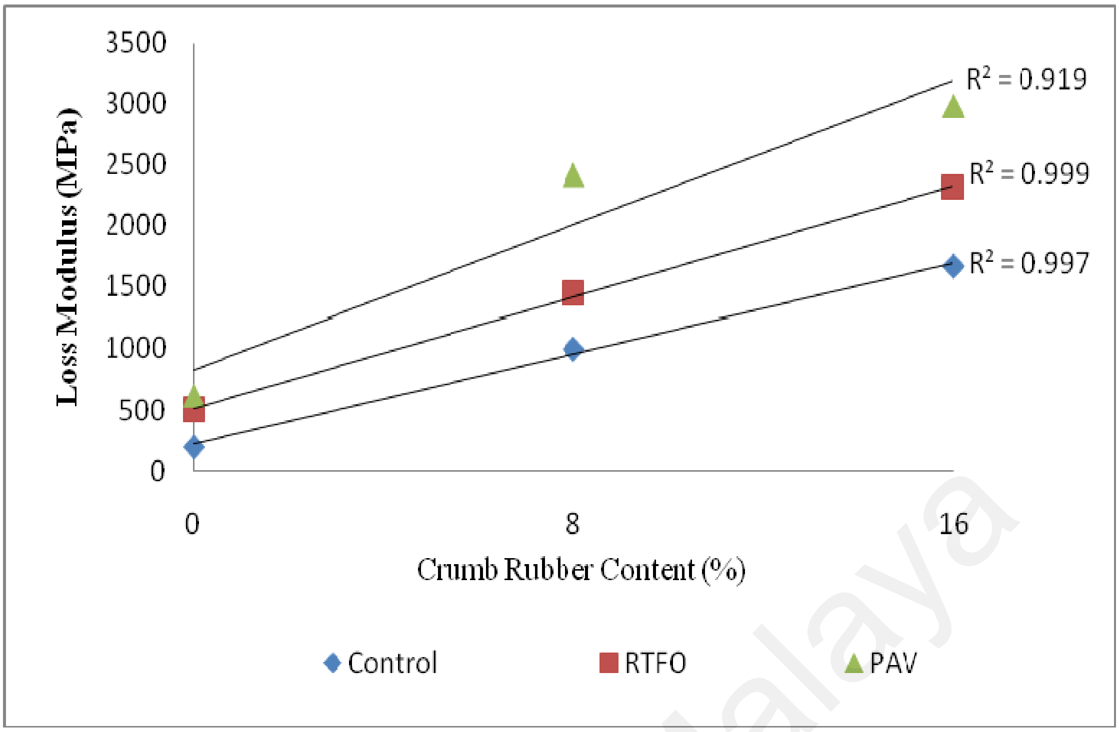


Figure 4.48: Loss Shear Modulus vs. Crumb Rubber Content of Specimens at 76˚C

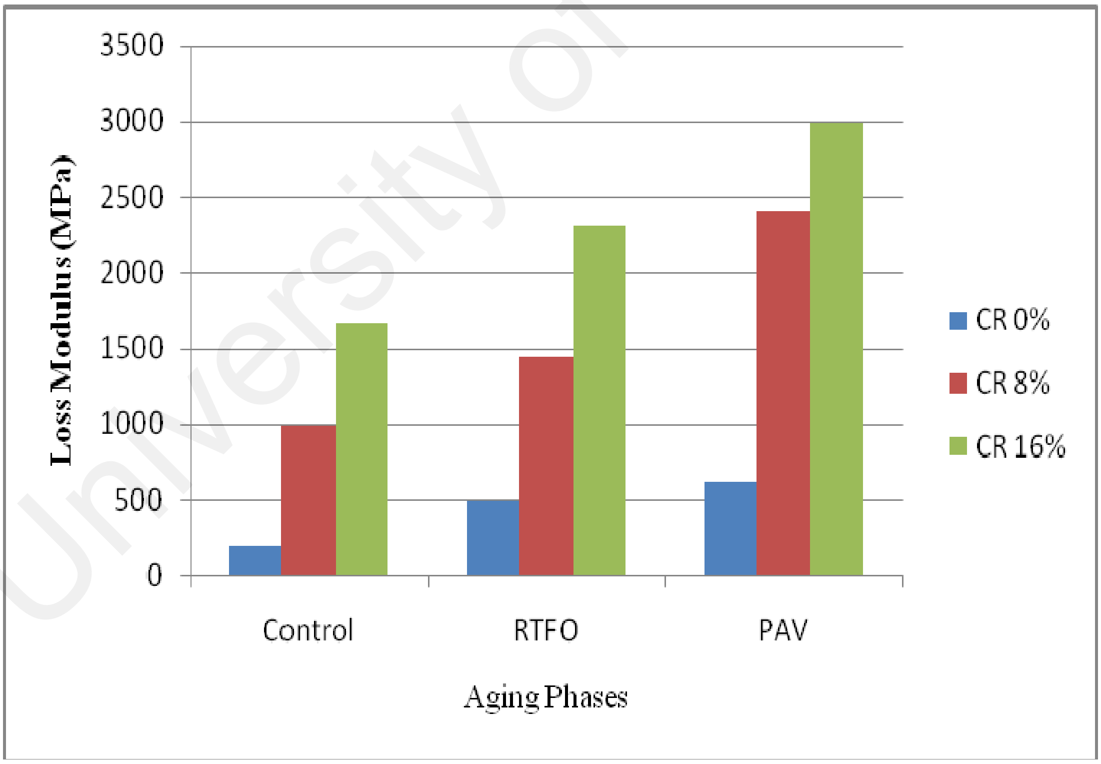


Figure 4.49: Loss Shear Modulus vs. Aging Conditions of Specimens at 76 ˚C

4.9 Analysis of Viscosity Results of Aged Rubberised Bitumen

Figures 4.50, 4.51 and Table 4.50 show the viscosity results at 135 °C for CRM binders with various rubber contents under different aging conditions. A general trend was found, as expected, which indicated that higher crumb rubber content would lead to higher viscosity for the CRM binders.

Table 4.51: Results of Viscosity Aged Rubberised Bitumen at 135˚C

Viscosity (MPa- s)				
Group	CRM	Mean	Std. Deviation	n
Control (unaged)	0%	405.3	15.01	3
	8%	2183.0	2.65	3
	16%	3386.7	2.52	3
RTFOT	0%	885.0	15.00	3
	8%	2437.3	11.68	3
	16%	2853.7	897.50	3
PAV	0%	1213.0	13.00	3
	8%	2893.0	6.00	3
	16%	4752.3	7.51	3

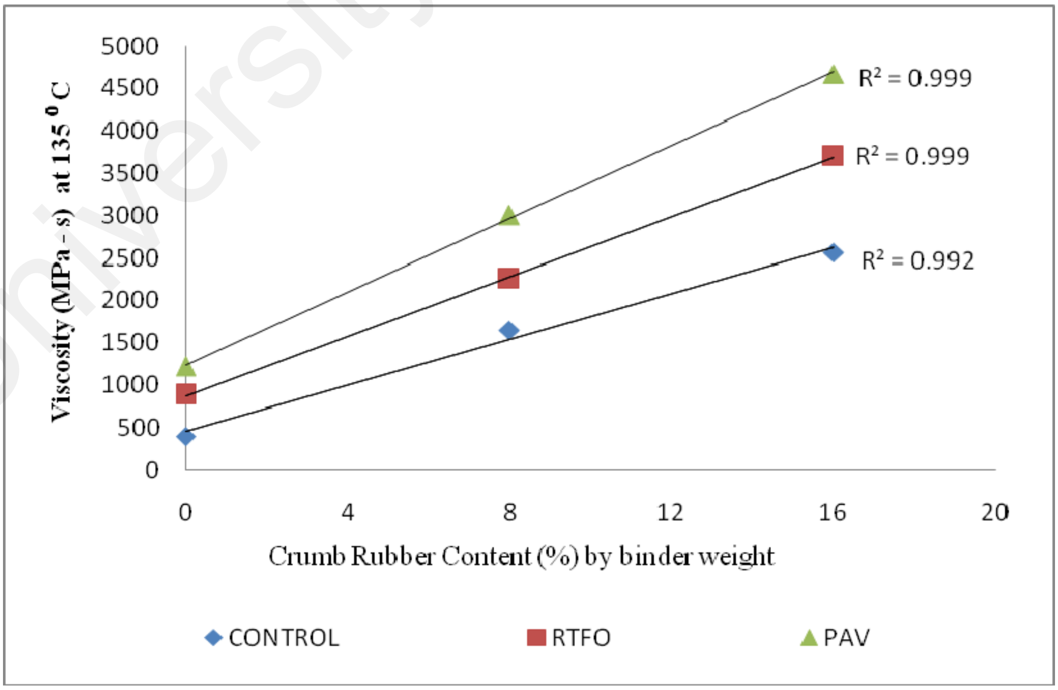


Figure 4.50: Brookfield Viscosity vs. Crumb Rubber Content of Specimens at 135˚C

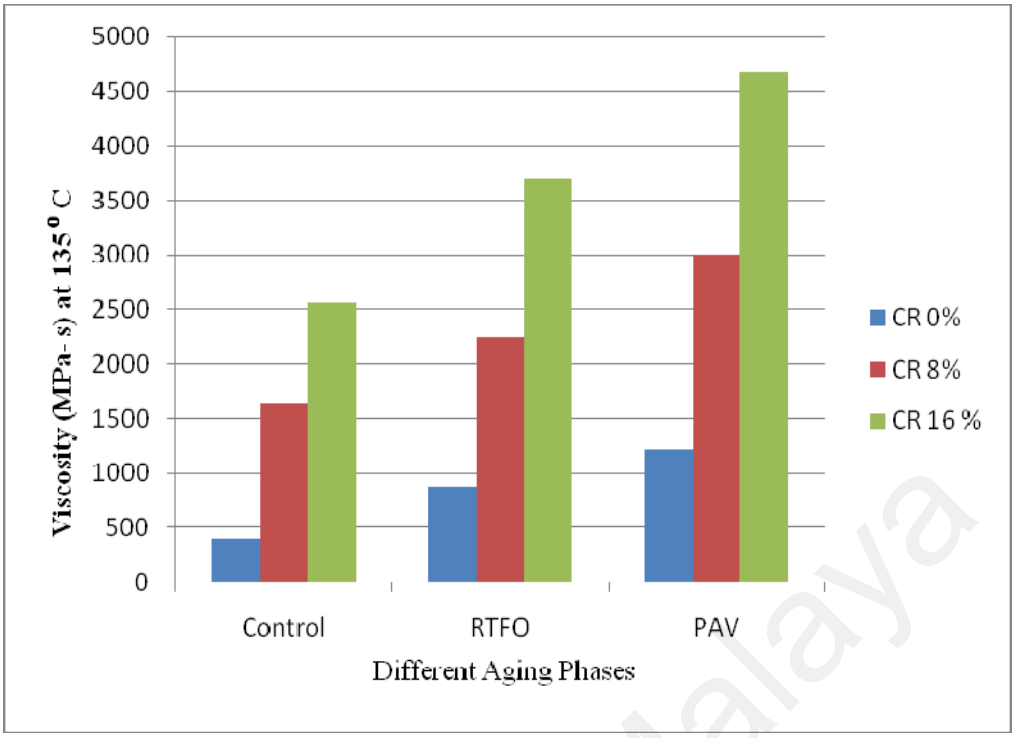


Figure 4.51: Brookfield Viscosity vs. Aging Phases of Specimens at 135˚C

Additionally, the increased viscosity of aged rubberised bitumen were about 36% and 44% for rubber contents of 8 % and 16% under RTFOT aging conditions, while the increase in the viscosity under PAV aging values were about 78% and 82% for rubber content of 8% and 16% respectively. Thus, the increase in crumb rubber content had an obvious effect on aged rubberised bitumen viscosity compared to aged unmodified bitumen with correlation coefficient $R^2 = 0.992$, 0.990 and 0.997 for unaged , RTFOT and PAV aging conditions respectively. This increased viscosity can be explained by the hardening of the bitumen binder due to evaporation of low molecular weight friction oil through aging (Mahrez, 1999).

Figure 4.52 shows VAI (viscosity aging index) of the original bitumen and the rubberised binder after RTFOT and PAV aging. VAI is the ratio of aged viscosity of the binder to the unaged viscosity of same binder as illustrated below:

VAI = V aged/ V unaged Eqn. 4.2

Where VAI is the viscosity aging index, V unaged and V aged are the viscosity of unaged and aged value of the binder respectively. The investigation exhibited that aging index of rubber modified binder is evidently lower than that of the original bitumen, indicating that the rubberised bitumen can dramatically improve the thermo- oxidative aging properties of bitumen. This result may be due to the chemical components change of the binder during aging that led to increase the asphaltene content which in turn was responsible for increasing the viscosity flow of the aged bitumen binder. The aging index of viscosity reflected better resistance of crumb rubber modified bitumen to aging as it show lower value after short – term aging ,RTFOT and long –term aging , PAV respectively. Thus, the use of viscosity index eliminates the variability caused by difference in viscosity of the original bitumen and modified bitumen which provides a more reliable picture of the hardening (aging) rate.

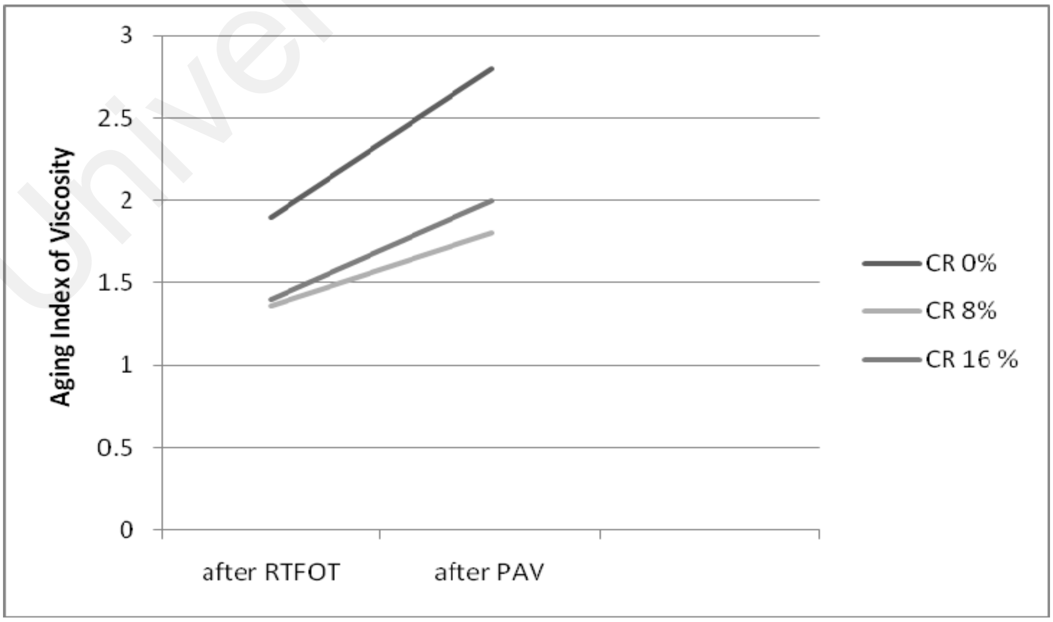


Figure 4.52: AIV of original and rubberised modified bitumen after RTFOT and PAV

Figures 4.53–4.55 and Tables 4.51–4.53 show the effect of temperature on unmodified bitumen and rubberised bitumen under various aging conditions. As temperature increased from 100 °C to 170 °C, the viscosity of rubberised bitumen reduced for samples with different CRM contents under RTFOT and PAV aging conditions, respectively. Moreover, the increase in rubber content improved the reduction in viscosity as temperature increased.

Bitumen aging occurred during mixing and construction processes, as well as during long-term aging. The increase in viscosity presented valuable effects on binder consistency and durability. Notably, based on the above mentioned results on viscosity, more improvements can be made; that is, by increasing rubber content as the main influencing factor. According to Huang (2008), as rubber content increases, rubber modification could improve rutting resistance because of the increase in viscosity and elasticity at high temperatures.

Table 4.52: Viscosity Results for Aged Unmodified Bitumen Only

Viscosity (MPa- s)							
Temperature C	**100**	**110**	**120**	**135**	**150**	**160**	**170**
Unaged	3712	1929	637	405	237	163	72
RTFOT	7670	4176	2050	885	503	299	137
PAV	13508	6745	3437	1213	817	664	420

Table 4.53: Viscosity Results for Aged Modified Samples with 8% Rubber Content

Viscosity (MPa- s)							
Temperature C	**100**	**110**	**120**	**135**	**150**	**160**	**170**
unaged	13094	6055	2200	2183	683	411	322
RTFOT	11250	7240	3110	2450	654	398	311
PAV	12545	10698	5680	2893	1094	766	589

Table 4.54: Viscosity Results for Aged modified Samples with 16% Rubber Content

Viscosity (MPa- s)							
Temperature C	**100**	**110**	**120**	**135**	**150**	**160**	**170**
unaged	1311	12200	5100	3386	930	679	388
RTFOT	12412	6399	5645	3890	1494	1191	888
PAV	24077	9820	6116	4752	2207	1215	1002

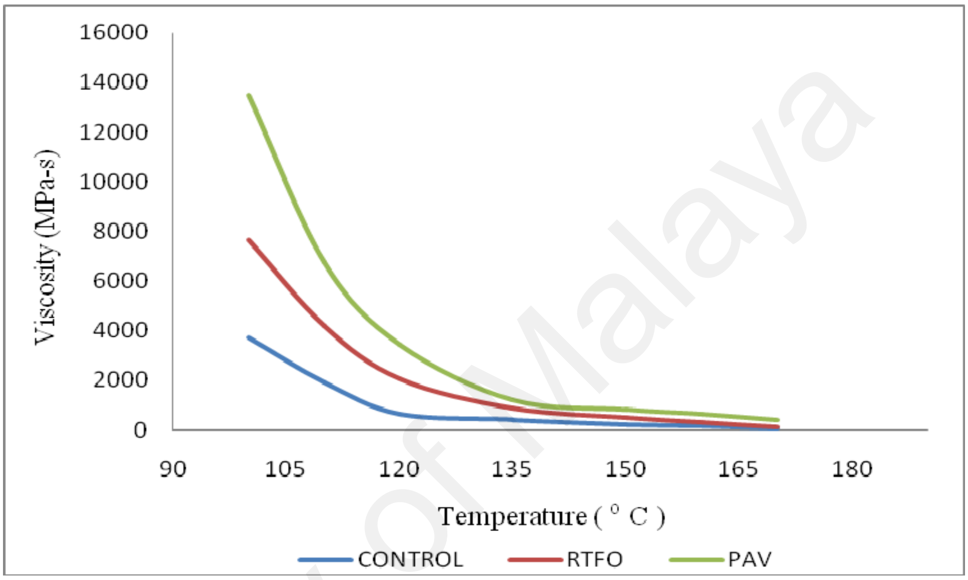


Figure 4.53: Brookfield Viscosity vs. Aging Conditions of Specimens at 0% Rubber Content

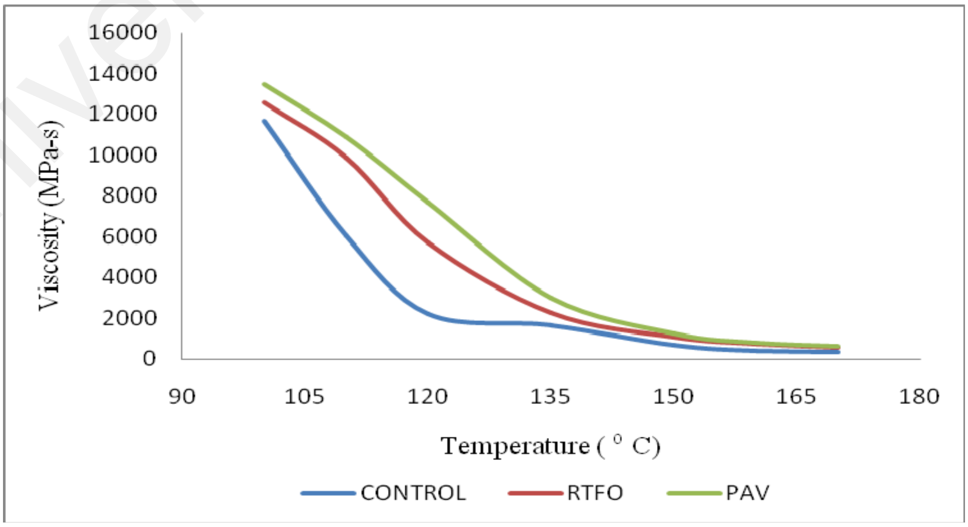


Figure 4.54: Brookfield Viscosity vs. Aging Conditions of Specimens at 8% Rubber Content

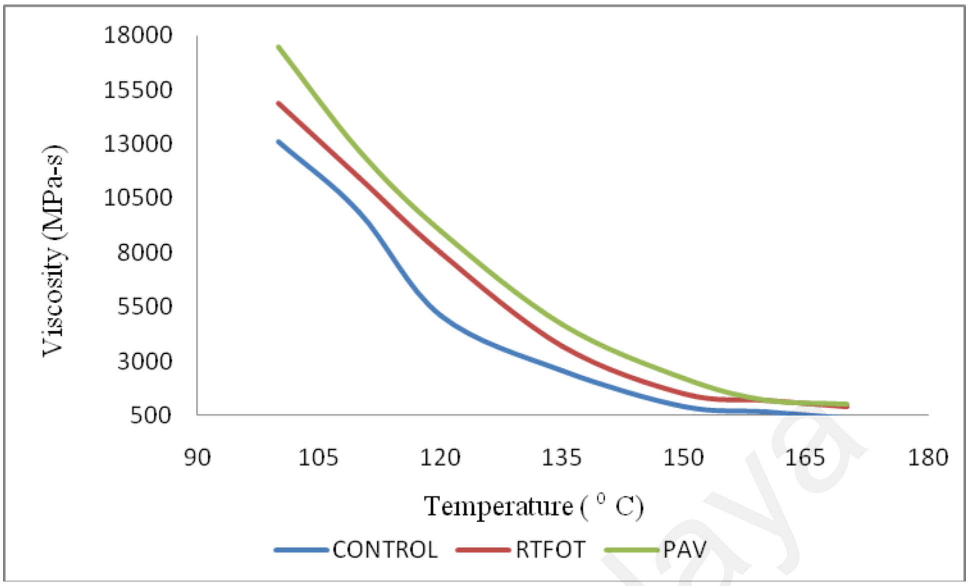


Figure 4.55: Brookfield Viscosity vs. Aging Conditions of Specimens at 16% Rubber Content

4.10 Analysis of Penetration Results of Rubberised Bitumen After Aging

Figures 4.56, 4.57 and Table 4.54 show the penetration results of modified and unmodified binder after aging. Based on the results, penetration decreased as crumb rubber content increased as shown in Figures 4.56 and 4.57. The penetration of the bitumen binder appears to dramatically decrease as the crumb rubber content increased with correlation coefficient of $R^2 = 0.999$, $R^2 = 0.996$ and $R^2 = 0.975$ for unaged, rolling thin file aging test (RTFOT) and pressure aging vessel aging (PAV), respectively.

Table 4.55: Results of Penetration of Aged Rubberised Bitumen at 25˚C

Penetration (0.1mm)				
Group	CRM	Mean	Std. Deviation	n
Control (unaged)	0%	84.0	2.00	3
	8%	60.7	2.08	3
	16%	40.0	1.00	3
RTFOT	0%	66.7	1.53	3
	8%	27.7	1.53	3
	16%	19.3	2.52	3
PAV	0%	43.0	2.00	3
	8%	22.0	2.00	3
	16%	9.7	2.52	3

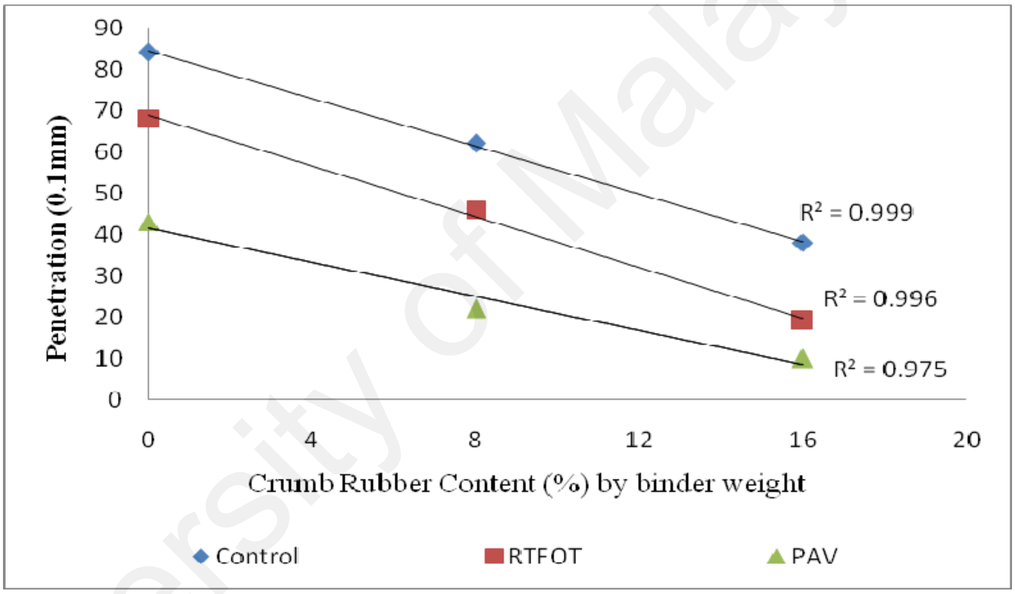


Figure 4.56: Penetration vs. CRM Content of Specimens at 25˚C

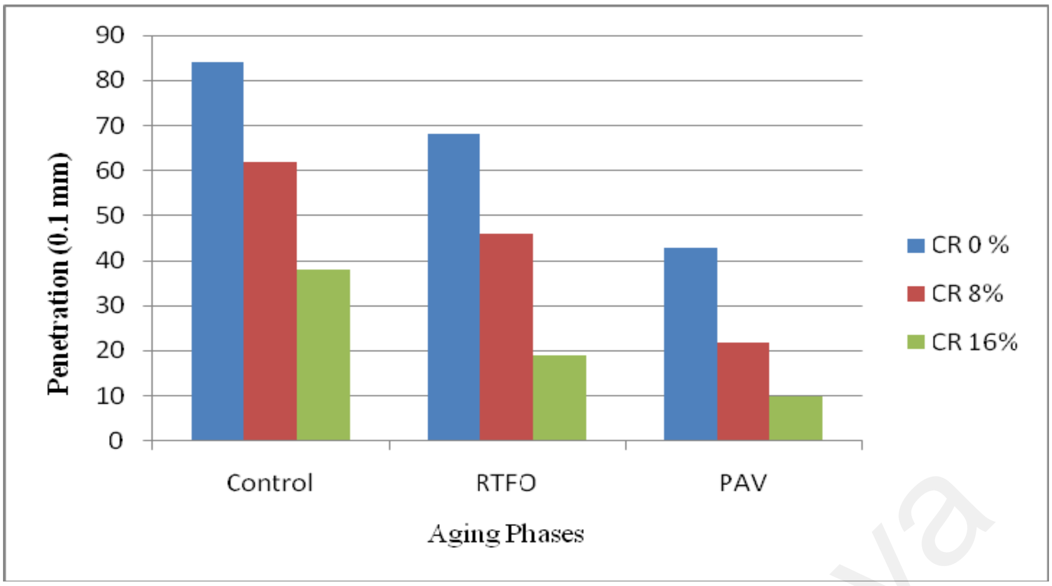


Figure 4.57: Penetration vs. Aging Phases of Specimens at 25˚C

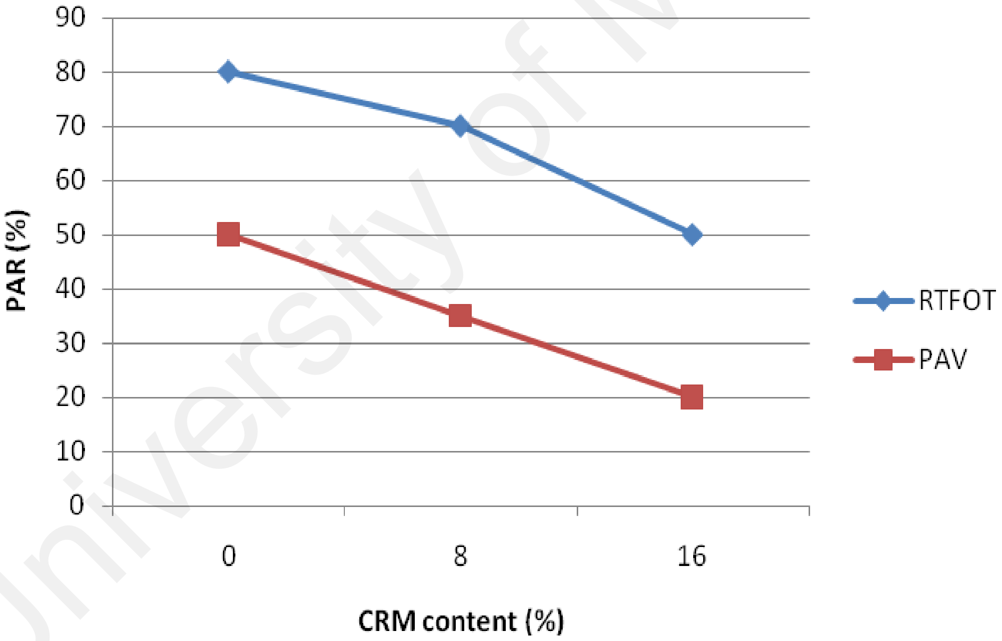


Figure 4.58: Effect of aging on penetration of bitumen binder

The penetration aging ratio can be perceived from another perspective to indicate the effect of aging on the physical properties of the bitumen binder (Cong *et al*., 2010). Penetration aging ratio (PAR) can be defined as the ratio of aged penetration value to unaged penetration as shown in Eq. 4.3

PAR = (aged penetration/ unaged penetration value) x 100 Eq. 4.3

Figure 4.58 shows the penetration aging ratio of original and rubberised bitumen after RTFOT, rolling thin film oven test and PAV, pressure aging vessel test. The results showed that the penetration of the original and rubberised bitumen binders decreased after the two different aging. In addition, the higher the rubber content, the lower penetration aging ratio led to reducing the degree of aging of rubberised bitumen binder. Meaning, the crumb rubber addition led to improved the binder resistance to oxidative aging.

4.11 Analysis of Softening Point of Rubberised Bitumen

Ring and ball softening point test was selected to determine the temperature at which the bitumen binder would become softer and more viscous. With gradual increase in temperature, the bituminous materials slowly changed from brittle to softer and with less viscous liquid flow.

Figures 4.59, 4.60 and Table 4.55 showed the effect of aging phases on the softening point for various crumb rubber contents. Results showed an increase in softening point with changes in aging conditions, rubber content increased for the 8% and 16% samples. Clearly, softening point increased as rubber content increased for both aging phases.

Table 4.56: Results of Softening Point of Aged Rubberised Bitumen at 25˚C

Softening point (ºC)				
Group	CRM	Mean	Std. Deviation	n
Control (unaged)	0%	48.0	0.71	3
	8%	51.0	1.41	3
	16%	60.5	1.41	3
RTFOT	0%	50.5	0.71	3
	8%	59.0	1.41	3
	16%	66.0	1.41	3
PAV	0%	56.5	0.71	3
	8%	63.5	2.12	3
	16%	69.5	2.12	3

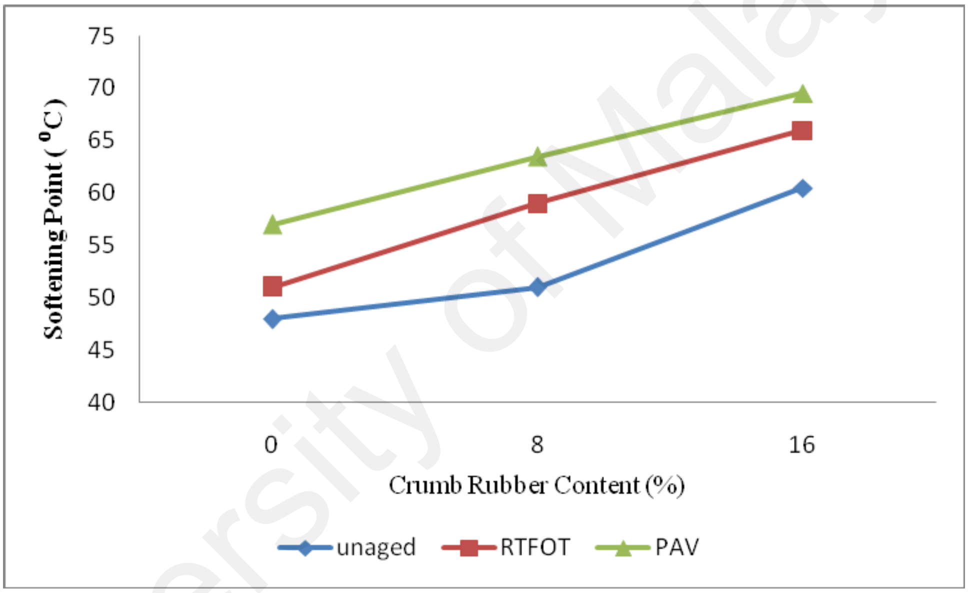


Figure 4.59: Softening Point vs. CRM Content of Specimens at 25˚C

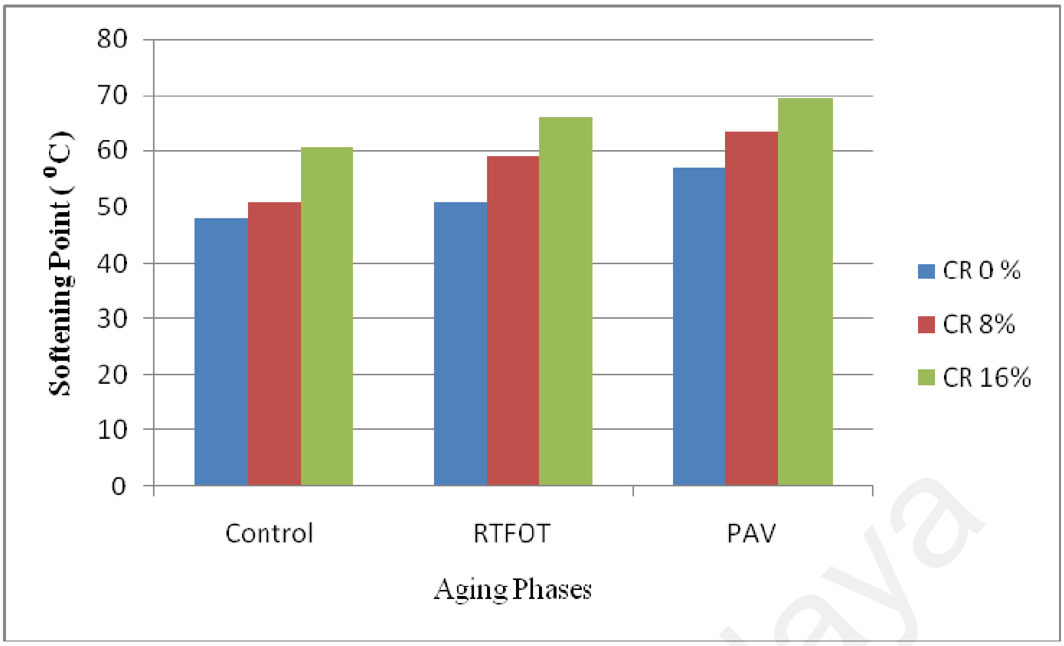


Figure 4.60: Softening Point vs. Aging Phases of Specimens at 25˚C

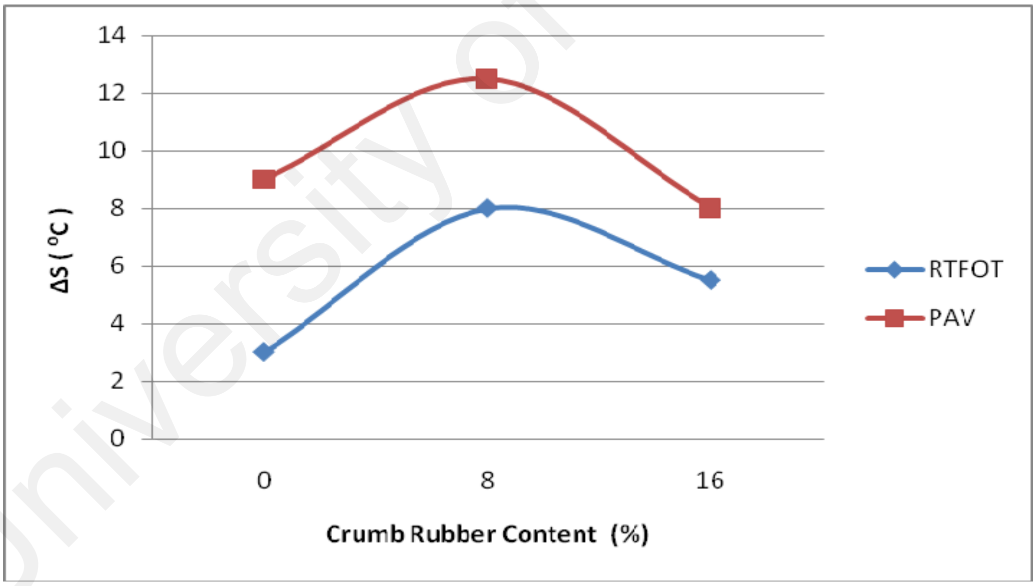


Figure 4.61: Effect of aging on softning point increment of bitumen binder

From Figures 4.59 and 4.60, it is evident that the crumb rubber with 8% and 16% rubber contents significantly affected softening point test, and these were higher than unmodified binder by about 1 to 1.5 times. The increase in ring and ball softening point was due to the oxidation reaction, which induced more asphaltene micelles. An increase in asphaltene with high molecular weight produced harder bitumen binders, as well as less susceptibility and higher resistance to pavement distress by shear force (e.g., rutting). Bitumen binders as function of aging conditions and crumb rubber content as evident in the current study, was also in tandem with the finding of Mahrez (1999). The softening point increment can be used as a good indicator to reflect the degree of aging and it can be defined by ΔS and calculated as in Eq. 4.4

$$\Delta S = S.P_{\text{aged}} - S.P_{\text{unaged}} \qquad \text{Eqn. 4.4}$$

Where ΔS is the softening point increment, S.P aged is aged softening point value and S.P unaged is the unaged softening point value. Figure 4.61 shows the effect of crumb rubber modified bitumen on softening point changes after RTFOT and PAV aging. The softening point results of original/unmodified bitumen and rubberised bitumen showed an increase after both aging methods of RTFOT and PAV respectively as rubber content was increased, indicating an inherent hardening process of the materials in aging. In comparing the softening point increment (ΔS) of modified binder with bitumen without crumb rubber modifier, the crumb rubber modified bitumen showed lower (ΔS) as rubber crumb content increased. This can be explained by the structural change of molecules in asphalt of fraction content during aging, which leads to an increase in the softening point (Cong *et al*., 2010). Thus the results of using rubberised bitumen binder have a significant effect on improving the aging resistance with lower hardening aging of the bitumen.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS FOR FUTHURE STUDIES

5.1 Conclusion

The main purpose of this study was to investigate the effect of blending conditions (blending temperature and blending time) on the performance properties of rubberised bitumen for different crumb rubber contents, as well as to study the effect of rheology behaviour of rubberised bitumen before and after aging.

From the analysis of the binder tests of rubberised bitumen, the researcher found that the performance binder properties exhibited significant advantages and could improve asphalt pavement resistance against permanent deformation rutting.

5.2 Effect of Blending Temperature

The results of binder tests on both unmodified and rubberised bitumen showed that blending temperature significantly affected the performance properties of rubberised bitumen compared with the unmodified bitumen. The increase in blending temperature corresponded to the increase in Brookfield viscosity, softening point, ductility, elastic recovery and complex shear modulus, as well as reduced penetration and phase angle. The increase in storage modulus was more obvious than for the loss modulus.

The two-way ANOVA showed clearly that blending temperature exhibited significant difference and influence on the performance properties of rubberised bitumen.

5.3 Effect of Blending Time

The results of binder tests on both unmodified and rubberised bitumen showed that blending time had no effect on the performance properties of rubberised bitumen and unmodified bitumen. The increase in blending time showed insignificant difference on rubberised bitumen properties in the case of 30 and 60 minutes. There was a very slight increase in Brookfield viscosity, softening point, ductility, elastic recovery and complex shear modulus; there was a very slight reduced penetration and slight reduction in phase angle. There was an extremely slight increase in both storage modulus and loss modulus. Based on the two-way ANOVA, blending time had insignificant difference on rubberised bitumen physical and rheological properties and a rather slight influence on the performance properties of rubberised bitumen.

5.4 Effect of Rubber Content on Rubberised Bitumen

According to laboratory binder tests, it is clear that rubber crumb content played a main role in influencing significantly the performance and rheological properties of rubberised bitumen binders. It could enhance the performance properties of asphalt pavement resistance against deformation during construction and road services.

The increase in rubber crumb content was from 4 - 20% thus indicating a liner increase in softening point, ductility, elastic recovery, viscosity, complex shear modulus and rutting factor. This phenomenon could be explained by the absorption of rubber particles with lighter fraction oil of bitumen, leading to increase in rubber particles during swelling during the blending process.

The increase in rubber content by 16% and 20% showed a corresponding increase in Brookfield viscosity value that are higher than SHRP specification limits (3 Pa). These make the two stated percentages unacceptable for field construction during asphalt pavement mixture construction.

5.5 Effect of Rheological properties of CRM Binders on Rutting

The rubberised bitumen binder with higher crumb rubber content has an obvious effect on the rheological properties (increase in complex shear modulus G*, storage modulus G’, loss modulus G’’ and decrease in phase angle δ). The results indicated that the rubber content has the potential to resist rutting deformation that occurs in road pavement as a result of increased traffic loading. The visco-elastic properties of rubberised bitumen exhibited an increase in storage modulus and decrease in phase angle, which were especially significant with lower increases in loss modulus. Furthermore, the results showed an increase in the elasticity of the modified binder and an enhanced ability to recover its original shape after stress removal.

5.6 Effect of Aging on Binder Performance

Results showed that aging affected binder durability and performance properties of bitumen binder. During the different aging conditions (RTFOT and PAV) and crumb rubber concentrations, both unmodified and rubberised bitumen presented a decrease in penetration, as well as increase in softening point and viscosity. These phenomena could be explained by the hardening of the binder due to the evaporation of the lower molecular weight oil fractions during aging processes. The rheological properties, (G*, G’ and G”) for all samples were increased after aging. Based on the results, crumb rubber content significantly improved durability of rubberised bitumen binder and led to higher resistance to aging. Thus, the crumb rubber modified bitumen led to less susceptible to permanent deformation.

5.7 Selection of the Proper Binder

The selection of proper materials and conditions should be based on certain criteria and standard equipment and specifications, such as improved resistance to rutting deformations.

Based on the analysis of tests results, a blending temperature of 180 °C and blending time of 30 min would be ideal for blending conditions. Additionally, rubberised bitumen with rubber crumb contents of 8%, 12%, and 16% could be used as candidate binders due to their better and higher quality.

5.8 Recommendations for Future Studies

Despite the different conclusions reached from this research project, a list of recommendations for future studies are summarised as follows:

- Further studies and more investigations should be conducted on crumb rubber-modified binder rheological properties and their effects under different mixing conditions.
- Performance properties related to pavement distresses, permanent deformations, rutting and fatigue should be evaluated.
- There is also a need to test more binder sources, different blending times of (90 200 and 240 min) and blending temperatures of (200, 230 and 300 °C) and various rubber particle sizes of (40 and 80 mesh).
- The rheological properties of rubberised bitumen under low-temperature conditions should be studied using the BBR test. More attention should be given to the chemical structure and physical properties of base bitumen.

- Further rheological characterisation of the studied materials should be carried out using different temperature degrees, different test geometries and configurations and other rheometers, in order to obtain the master curves, the transition temperatures (Tg) and the fatigue resistance of the binders.

- RTFOT aging procedure of rubber particles continue to interact with bitumen. This phenomenon induces additional changes in the morphology of the rubber particles and in the bitumen. This should be further investigated in aged rubberised bitumen binders and in residual bitumen**.**

- The rubberised bitumen production is largely controlled by physical factors such as temperature, digestion time and stirring velocity. The influence of these factors in the final characteristics of the binders and in the dimension of the swelled rubber particles should also be evaluated;

- The conclusions of this work show that the use of softer bitumen develops binders with an improved behaviour in flexible road pavements. However, these conclusions were only obtained through binder characterisation. Therefore, these results should be validated by carrying out tests in bituminous mixtures and by evaluating pavement trials constructed with this rubberised bitumen.

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APPENDIX A : DSR RESULS OF DIFFERENT BLENDING TEMPERATURE AND BLENDING TIME

Table TA1: DSR Results of Unmodified Samples at Blending Temperatures of 160˚C and 30 minutes Blending Time

DSR				
T(C˚)	**G'[pa]**	**G" [pa]**	**G*[pa]**	**δ[˚]**
31.4	10420	316000	326420	71.6
33.4	4700	193000	197700	75
35.5	6021	122500	128521	77.6
37.5	2342	80490	82832	79.5
39.5	2628	55540	58168	81
41.6	1986	39820	41806	82.2
43.6	1509	30220	31729	83.1
45.7	792	22290	23082	83.9
47.4	684	15680	16364	84.8
49.7	426	11050	11476	85.5
51.3	280	7909	8189	86.1
53.9	200	5733	5933	86.7
55.8	129	4185	4314	87.2
58	123	3099	3222	87.6
60	118	2319	2437	87.9
62	65	1740	1805	88.3
64	37	1313	1350	88.6
66	33	1005	1038	88.8
68	23	593	616	89
70.2	10.7	462	473	89.1
72	10.2	366	376	89.2
74	9.8	290	297	89.01
76	9.6	229	235	89.23
78	8.9	184	184	89.61
80	7.88	175	180	89.31

Table TA2: DSR Results of Unmodified Samples at Blending Temperature of 160˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31.4	92640	288700	381340	72.2
33.4	33970	134500	168470	75.8
35.5	17680	79140	96820	77
37.5	10740	52470	63210	78.4
39.5	6873	36560	43433	79.3
41.6	4717	26850	31567	80
43.6	3192	20150	23342	81
45.7	2068	15090	17158	82
47.4	1291	11000	12291	83
49.7	787	8240	9027	84.5
51.3	462	6316	6778	85.8
53.9	301	5010	5311	86.7
55.8	198	4020	4218	87
58	131	3152	3283	87.6
60	84	2439	2523	88
62	54	1842	1896	88.3
64	34	1395	1429	88.5
66	21	1058	1079	88.8
68	14	810	824	88.9
70.2	13.7	622	632	89.2
72	13	482	488	89.7
74	12.88	377	377	89.73
76	12.12	297	297	89.67
78	10.3	236	236	89.54
80	9.8	188	193	89.22

Table TA3: DSR Results of Unmodified Samples at Blending Temperature of 180˚C and 30 minutes Blending Time

T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31.4	95520	271600	367120	70.62
33.4	52230	172600	224830	73.17
35.5	30310	115100	145410	75.25
37.5	18290	79520	97810	77.05
39.5	11300	56930	68230	78.77
41.6	7259	42190	49449	80.24
43.6	4936	33290	38226	81.57
45.7	2908	23960	26868	83.08
47.4	1662	17240	18902	84.5
49.7	964.6	12330	13294.6	85.5
51.3	562	8842	9404	86.3
53.9	344.9	6390	6734.9	86.9
55.8	211	4614	4825	87.38
58	133.6	3404	3537.6	87.7
60	82.67	2526	2608.6	88.13
62	52.17	1889	1941	88.42
64	32.9	1417	1449.9	88.6
66	20.8	1070	1090.8	88.8
68	18	813	826.5	89
70.2	16.88	627.9	637	89.15
72	14	486	491	89.33
74	14.5	382	387	89.4
76	13.45	303	308	89.67
78	13	239.6	244	89.88
80	12.88	193.7	198	89.91

Table TA4 : DSR Results of Unmodified Samples at Blending Temperature of 180˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31.15	89210	273000	262210	71.9
33.26	44710	169300	214010	75.2
35.35	23610	107100	130710	77.5
37.41	13160	69980	83140	79.3
39.48	7631	47160	54791	80.8
41.52	4715	33600	38315	82
43.57	3000	24730	30730	83
45.63	1961	18440	20401	83.9
47.65	1216	13470	14686	84.8
49.72	761	9839	10600	85.5
51.75	483	7230	7713	86.1
53.81	293	5212	5502	86.7
55.85	184	3796	3980	87.2
57.92	114	2822	2936	87.6
59.9	73	2123	2196	88
61.98	48	1603	1651	88.2
64	30	1214	1244	88.5
66	20	925	945	88.7
68	19.67	709	722	88.9
70.1	17.88	546	555	89
72.1	16.7	425	431	89.1
74.2	15	333	333	89.44
76.2	14.1	262	262	89.67
78.3	12	206	206	89.88
80.4	12	194	200	89.88

Table TA5: DSR Results of Modified Samples of 4% rubber at Blending Temperature of 160˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	35900	120600	156500	65
33	33100	118660	151760	67
35	27500	112090	139590	68
37	25999	100900	124899	70
39	22111	98450	118561	72
41	20888	90800	108688	73
43	17090	67343	82433	74
45	15780	55232	68,012	78
47	10888	33110	42,998	80
49	8780	20000	26,780	82
51	7555	14890	19,445	83
53	5454	10090	13,544	85
55	3212	7689	10,901	85.8
57	2450	5670	8,120	86.5
59	2090	3444	5,534	86.9
61	1890	3000	4,890	87
63	1400	2890	4,090	87.3
65	970	2100	2,770	87.5
68	720	1567	1,987	87.7
70	200	1200	1201	87.9
72	67	1100	1100	88.3
74	34	1050	1501	88.4
76	44	700	755	88.5
78	22	550	593	88.5
80	12	450	482	89

Table TA6: DSR Results of Modified Samples of 4% rubber at Blending Temperature of 160˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	58320	170000	228,320	62
33	55890	160,450	216,340	71
35	51234	149,211	200445	73.5
37	48567	138,760	187,327	75.6
39	42333	133,900	176233	77
41	40000	128,444	168,444	78.3
43	38909	122,434	161,343	79.5
45	35660	118,888	154,548	80.5
47	31988	113,200	145,188	81.4
49	28769	111,100	139,869	82.5
51	24540	90,789	115,329	83.4
53	20100	75,343	95,443	83.6
55	16900	50,202	67102	84.6
57	14540	35,012	49,552	85.5
59	9000	22,100	31,100	85.7
61	4300	15,900	20200	86.4
63	2134	7,600	9734	86.7
66	1090	4309	5399	86.9
68	880	3100	3980	87.4
70	300	1400	1401	87.6
72	230	1330	1330	87.8
74	120	1200	1201	87.9
76	79	888	889	88
78	45	506	507	88
80	39	489	490	88.5

Table TA7 : DSR Results of Modified Samples of 4% rubber at Blending Temperature of 180˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31.1	62400	171500	233900	62.33
33.2	61200	150909	212109	64.8
35.3	58990	145787	204777	65.9
37.4	56089	133900	189989	66.8
39.4	53787	121878	175665	67.9
41.5	51909	120100	172009	68.99
43.5	48545	88909	137454	70
45	44900	55454	100354	72.6
47.6	21010	34888	55898	74.33
49.6	10909	22343	33252	75.8
51.7	7980	20999	28979	76.9
53.6	5121	17656	22777	77
55.7	4323	13545	17868	78.9
57.8	2100	10900	13000	80
59.8	1989	8555	10544	80
61.9	878	5444	6322	81.5
63.9	676	4090	4766	81.9
65.9	623	3450	4073	82.5
68	600	2890	3490	83.4
70	250	1550	1560	83.5
72.3	234	1342	1349	83.8
74.7	178	1280	1286	84.3
76	90	998	1001	84.9
78.6	67	670	672	85.8
80	44	530	531	86.66

Table TA 8 : DSR Results of Modified Samples of 4% rubber at Blending Temperature of 180˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	77900	188900	266800	70
33	75676	185434	261110	71.5
35	71090	180200	251290	71.9
37	68456	175444	243900	72.6
39	64989	168454	233443	73.5
41	56232	159888	216120	73.9
43	51088	140900	191988	74.6
45	47121	133767	180888	74.9
47	42720	120100	162820	75.7
49	37212	100333	137545	76
51	26555	66454	93009	77.5
53	18090	43100	61190	77.8
55	12670	33100	45770	78.6
57	10100	22898	32998	78.9
59	8565	17232	25797	79.5
61	6343	15222	21565	79.99
63	3010	11900	14910	80.7
66	2134	8900	11034	81.6
68	998	5100	6098	81.98
70	290	1590	1603	82.54
72	254	1370	1380	82.88
74	213	1188	1195	83.5
76	100	1000	1005	83.99
78	88	780	784	84.23
80	50	577	579	84.98

Table TA 9 : DSR Results of Modified Samples of 8 % rubber at Blending Temperature of 160˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G" [pa]**	**G* [pa]**	**δ[˚]**
31	81090	191200	272290	61.23
33	79200	181900	261100	61.45
35	77545	178343	255888	61.77
37	68900	162100	231000	61.88
39	66212	155434	221646	62.65
41	61232	142660	203892	64.8
43	57878	120000	177878	65.99
45	53201	110989	164190	66.45
47	48900	87121	136021	67
49	43878	67232	111110	68.9
51	36100	55678	91778	69.9
53	21787	48999	70786	70.4
55	14000	32199	46199	71.6
58	8323	28900	37132	72.55
60	5100	17450	22550	74.56
62	2134	12898	15032	76.9
64	1909	7888	9797	77.89
66	845	5989	6834	78.56
68	700	4477	5177	79.8
70	400	1889	1918	80.01
72	333	1760	1781	80.99
74	212	1555	1572	81.55
76	140	1300	1313	81.89
78	100	1090	1100	82.22
80	77	800	806	82.77

Table TA 10 : DSR Results of Modified Samples of 8 % rubber at Blending Temperature of 160˚C and 60 minutes Blending Time

DSR				
T (C˚)	G' [pa]	G'' [pa]	G* [pa]	δ[˚]
31	80100	212100	292200	65.4
33	77888	210989	288877	66.9
35	74121	188670	262791	67.5
37	66400	179454	245854	67.9
39	60787	172100	232887	68.6
41	54232	169500	223732	69.99
43	50100	161100	211200	70
45	44232	157430	201662	70.4
47	38670	149760	188430	70.99
49	20200	133100	153300	71.8
51	16430	121900	138330	72.6
53	10898	115676	126574	72.98
55	5343	110100	115443	73.5
58	4100	88900	93000	74.7
60	3333	40670	44003	75.2
62	2010	20134	22144	75.88
64	1880	12454	14334	76.1
66	900	7650	8550	76.45
68	870	5340	6210	77.8
70	490	1915	1956	78.2
72	560	1760	1796	78.5
74	340	1544	1574	78.87
76	210	1430	1452	79.88
78	170	1101	1118	79.99
80	90	898	911	80.24

Table TA 11: DSR Results of Modified Samples of 8 % rubber at Blending Temperature of 180˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	83200	225300	250244	64.2
33	80787	220900	244175	64.78
35	77340	190450	209295	65.5
37	71300	181200	197438	66.6
39	65400	176500	190496	67.9
41	61230	155434	166604	68.9
43	58200	140200	149292	69.9
45	47310	122900	130787	70
47	40100	116780	123809	70.6
49	35670	110560	117244	70.56
51	27590	88450	93269	71.5
53	19800	55450	58343	71.88
55	11230	33120	34826	71.99
58	7400	26890	28202	72.45
60	5230	15900	16559	73.77
62	3010	12780	13296	73.98
64	2100	10340	10744	74.23
66	1890	7800	8084	74.77
68	900	4230	4379	75
70	570	1967	2035	75.11
72	611	1780	1838	75.54
74	530	1640	1683	76.88
76	298	1570	1608	77.5
78	211	1256	1281	78.5
80	118	920	937	78.88

Table TA 12: DSR Results of Modified Samples of 8 % rubber at Blending Temperature of 180˚C and 60 minutes Blending Time

DSR				
T (C°)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	80100	228900	261461	61.1
33	71343	221898	250277	61.45
35	65900	180767	202165	62
37	52676	160343	181247	62.21
39	48666	120300	135562	62.55
41	42101	113888	126950	63.78
43	36550	100450	111770	63.99
45	30200	88400	96848	65.89
47	25434	76454	83330	66.56
49	16300	44232	47897	67.44
51	11222	35900	38689	68.11
53	7565	28767	30636	69.88
55	5090	22100	23518	70
58	4500	17232	18237	70.89
60	3670	12090	12769	71.23
62	3100	7100	7474	71.78
64	2898	4232	4450	71.99
66	1870	3090	3238	72.56
68	1020	2898	3023	73.45
70	660	1995	2075	73.99
72	600	1950	2025	74.32
74	591	1789	1856	74.5
76	355	1788	1842	75.99
78	321	1300	1336	76.54
80	143	967	992	76.89

Table TA 13 : DSR Results of Modified Samples of 12 % rubber at Blending Temperature of 160˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	79320	198000	231972	55.5
33	66450	196220	231631	58.7
35	60100	188790	220271	58.99
37	54390	166454	192398	59.9
39	49200	144300	166123	60.3
41	38767	139000	159281	60.77
43	27409	133200	150872	61.99
45	22900	127090	143278	62.5
47	17121	117680	132216	62.88
49	13454	110343	123243	63.55
51	10111	78290	86546	64.77
53	6232	45112	49779	64.99
55	4390	28100	30922	65.33
58	3190	15222	16665	65.98
60	2899	10900	11930	66.01
62	2450	7100	7752	66.33
64	1800	5340	5782	67.45
66	1450	3888	4193	68
68	1201	3234	3445	69.8
70	743	2020	2136	71
72	659	1867	1964	71.88
74	677	1820	1096	72.66
76	600	1798	1872	73.8
78	310	1490	1544	74.8
80	169	1100	1136	75.7

Table TA 14 : DSR Results of Modified Samples of 12 % rubber at

Blending Temperature of 160˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	59880	175300	204725	58.9
33	44567	168900	195622	59.7
35	36200	158200	182692	59.99
37	25100	140000	161301	60.22
39	20212	132560	151577	60.99
41	16343	124300	141373	61.55
43	10900	118100	133905	61.88
45	8200	110790	124875	62.54
47	6212	77400	86122	63.99
49	5300	54680	60611	64.44
51	5000	33120	36612	64.77
53	4230	27100	29904	64.99
55	3898	20900	22958	65.55
58	3100	17300	18938	65.99
60	2900	14232	15565	66.11
62	2780	10009	10892	66.77
64	2100	8434	9163	66.99
66	1300	6200	6687	67.99
68	1001	4100	4401	68.66
70	790	2150	2299	69.22
72	760	2000	2115	70.99
74	688	1945	2053	71.33
76	644	1830	1925	71.88
78	290	1690	1773	72.33
80	180	1230	1287	72.76

Table TA 15: DSR Results of Modified Samples of 12 % rubber at Blending Temperature of 180˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	97600	280100	343201	54.7
33	77909	255210	308202	55.9
35	51300	200000	240678	56.2
37	46100	193900	231225	56.99
39	31676	118200	140461	57.3
41	22343	100100	118048	57.99
43	15212	77340	90755	58.45
45	12090	50210	58243	59.55
47	10900	34111	39391	59.99
49	8232	27909	32194	60.1
51	6540	20454	23438	60.34
53	6200	16222	18587	60.78
55	5400	10999	12480	61.8
58	4888	8121	9198	61.99
60	4309	7300	8200	62.9
62	3500	6209	6931	63.6
64	2300	5100	5631	64.9
66	1945	4010	4393	65.88
68	1340	3680	4000	66.9
70	800	2166	2336	68
72	760	2010	2145	69.5
74	724	1966	2093	69.9
76	710	1910	2011	71.7
78	680	1650	1735	71.99
80	340	1444	1518	72

Table TA 16 : DSR Results of Modified Samples of 12 % rubber at Blending Temperature of 180˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	91200	252600	310252	52.3
33	88232	210900	261018	53.9
35	77100	188670	233208	54
37	55787	156300	188977	55.8
39	39111	144232	173995	55.99
41	26450	120989	144279	56.45
43	19300	115900	138210	56.99
45	12676	110343	130399	57.8
47	9400	92101	107788	58.7
49	8232	88400	102915	59.2
51	7660	66500	77139	59.55
53	6400	40343	46588	59.99
55	6000	31000	35756	60.11
58	5300	23100	26285	61.5
60	4222	18100	20332	62.9
62	3100	11200	12514	63.5
64	2190	7200	7970	64.6
66	2018	5400	5939	65.4
68	1888	3211	3505	66.34
70	835	2189	2386	66.5
72	800	2120	2288	67.9
74	766	1988	2137	68.45
76	730	1945	2069	69.99
78	688	1767	1876	70.34
80	360	1499	1589	70.55

Table TA 17 : DSR Results of Modified Samples of 16 % rubber at Blending Temperature of 160˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	50530	129600	157257	55.5
33	44900	124900	150834	55.9
35	35676	117656	140480	56.88
37	27989	111899	132093	57.9
39	16500	88700	104096	58.44
41	10450	72450	84531	58.99
43	8432	58340	67743	59.45
45	7909	41989	48489	59.99
47	5121	32767	37836	60
49	4888	20100	23139	60.33
51	4325	16290	18665	60.78
53	3523	11450	13063	61.22
55	2432	9888	11256	61.45
58	2100	7432	8418	61.99
60	1878	6450	7261	62.65
62	1640	5555	6240	62.89
64	1020	4190	4688	63.33
66	888	3200	3557	64.11
68	864	2760	3051	64.77
70	850	2200	2427	65
72	790	2090	2297	65.45
74	766	2061	2244	66.7
76	751	1977	2135	67.8
78	541	1840	1984	67.99
80	400	1522	1635	68.5

Table TA 18: DSR Results of Modified Samples of 16 % rubber at Blending Temperature of 160˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	64000	154600	180741	58.8
33	54300	150444	175697	58.9
35	48767	146333	169832	59.5
37	42100	139888	161855	59.8
39	37600	132000	152435	59.99
41	30989	125900	145376	60
43	24300	120434	138606	60.33
45	20767	116555	133419	60.88
47	17433	110111	126030	60.89
49	12909	87600	100157	61
51	10232	55400	63075	61.44
53	8450	34122	38724	61.78
55	7333	28676	32480	61.99
58	6212	20090	22753	62
60	5444	17400	19666	62.22
62	4200	11560	13038	62.45
64	3100	8232	9249	62.88
66	2000	6300	7070	63
68	1670	4011	4483	63.45
70	867	2255	2523	63.66
72	890	2167	2411	63.98
74	734	1900	2102	64.67
76	766	1989	2185	65.5
78	620	1856	2033	65.89
80	432	1580	1726	66.22

Table TA 19: DSR Results of Modified Samples of 16 % rubber at Blending Temperature of 180˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	67740	167700	213163	51.88
33	58777	158900	201045	52.22
35	47690	144200	180582	52.99
37	39000	135900	169919	53.11
39	31689	123676	153105	53.88
41	26545	110989	136621	54.33
43	20989	100100	122364	54.89
45	17565	87656	105869	55.89
47	11232	67545	81483	55.99
49	9777	45323	54529	56.22
51	8555	31200	37252	56.88
53	7434	27656	32979	56.99
55	6454	19232	22735	57.77
58	5900	12300	14507	57.98
60	4323	10888	12762	58.55
62	4100	8676	10122	58.99
64	3689	5232	6075	59.45
66	2767	4100	4735	59.98
68	1564	3454	3957	60.78
70	870	2355	2692	61
72	890	2190	2493	61.45
74	783	2088	2345	62.89
76	775	2003	2232	63.78
78	589	1910	2111	64.78
80	488	1650	1811	65.21

Table TA 20: DSR Results of Modified Samples of 16 % rubber at Blending Temperature of 180˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	69330	168800	211110	53.09
33	58900	177555	220395	53.67
35	47300	160900	198908	53.99
37	32900	154232	189095	54.65
39	28100	144200	176057	54.99
41	20888	135800	165118	55.33
43	17222	126100	152699	55.67
45	10989	110900	133801	55.98
47	9666	88777	106339	56.6
49	8343	65333	77998	56.89
51	7121	44900	53543	56.99
53	6000	38100	45255	57.34
55	5320	23190	27351	57.88
58	4100	18666	22015	57.98
60	3900	12900	15121	58.55
62	2888	10500	12252	58.98
64	1922	8565	9993	58.99
66	1721	6444	7483	59.44
68	1090	3111	3600	59.78
70	884	2380	2748	60.33
72	820	2210	2535	60.65
74	799	2100	2401	61.89
76	781	2022	2292	62.24
78	610	1977	2225	62.67
80	590	1780	1999	62.89

Table TA 21: DSR Results of Modified Samples of 20 % rubber at Blending Temperature of 160˚C and 30 minutes Blending Time

DSR				
T (C°)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	80620	179500	221258	54.22
33	71222	185343	226902	54.77
35	62343	173444	211144	55.23
37	55444	161200	194972	55.77
39	42100	155999	188413	55.89
41	36900	141200	169641	56.34
43	24333	137656	164566	56.77
45	16545	129000	153832	56.99
47	10111	120900	143445	57.44
49	8454	111787	132134	57.78
51	7343	88545	104422	57.99
53	6700	55900	65600	58.21
55	5111	43121	50412	58.88
58	4232	38111	44466	58.99
60	3109	21999	25605	59.22
62	2454	11898	13772	59.76
64	2100	9121	10534	59.98
66	1878	7010	8066	60.34
68	1200	5232	5988	60.89
70	892	2391	2727	61.22
72	844	2200	2496	61.78
74	800	2090	2369	61.89
76	799	2110	2423	60.55
78	712	1980	2266	60.89
80	610	1812	2071	60.99

Table TA 22 : DSR Results of Modified Samples of 20 % rubber at Blending Temperature of 160˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	97600	188000	232706	53.89
33	90888	176555	217628	54.22
35	86555	165900	202576	54.98
37	71232	144565	175521	55.45
39	63100	134900	163161	55.77
41	54000	129000	155620	55.99
43	45100	120989	145393	56.32
45	38900	115444	137666	56.99
47	26787	92343	109698	57.33
49	20900	76000	89844	57.77
51	17690	54333	64075	57.99
53	10999	32222	37950	58.11
55	8676	28090	32888	58.66
58	7454	20999	24503	58.98
60	6222	17454	20267	59.45
62	5100	10333	11946	59.88
64	4232	7500	8641	60.22
66	2999	5909	6757	60.98
68	1980	3121	3653	61.55
70	923	2399	2717	61.99
72	911	2211	2573	59.22
74	8450	2099	2429	59.76
76	821	2132	2467	59.77
78	700	2081	2403	59.99
80	622	1900	2235	58.22

Table TA 23: DSR Results of Modified Samples of 20 % rubber at Blending Temperature of 180˚C and 30 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	66020	141200	176292	53.22
33	56434	136788	169793	53.67
35	54232	122100	150943	53.99
37	40100	116444	143335	54.33
39	38900	110100	134588	54.89
41	26111	98777	120746	54.99
43	20100	77656	94048	55.66
45	18700	54333	65802	55.88
47	14200	38767	46516	56.45
49	10900	30999	36966	56.99
51	8100	26555	31503	57.45
53	7656	18676	22024	57.99
55	6333	10999	12930	58.22
58	5111	8545	10027	58.45
60	4000	7323	8563	58.77
62	3444	5333	6222	58.98
64	3100	4121	4791	59.33
66	2909	3222	3732	59.67
68	1898	2100	2427	59.88
70	951	2412	2788	59.89
72	910	2311	2668	59.99
74	899	2211	2597	60
76	833	2199	2406	60.77
78	769	2090	2443	60.88
80	631	1988	2319	61.11

Table TA 24: DSR Results of Modified Samples of 20 % rubber at Blending Temperature of 180˚C and 60 minutes Blending Time

DSR				
T (C˚)	**G' [pa]**	**G'' [pa]**	**G* [pa]**	**δ[˚]**
31	77240	197900	250389	52.22
33	68555	181200	227246	52.88
35	58900	172333	215841	52.98
37	45000	155400	193468	53.44
39	34888	132000	163870	53.66
41	26999	128777	158955	54.11
43	20999	118454	145211	54.66
45	165500	112900	137842	54.99
47	112900	95343	116081	55.22
49	9454	65343	79145	55.65
51	8333	43222	52209	55.88
53	7222	31000	37397	55.99
55	6000	23100	27792	56.22
58	5111	17600	21123	56.43
60	3555	11900	14244	56.66
62	2999	7600	9074	56.88
64	1888	4000	4763	57.11
66	1540	3222	3818	57.55
68	1094	2888	3409	57.88
70	967	2422	2845	58.34
72	933	2311	2705	58.66
74	894	2210	2578	58.99
76	877	2200	2566	59
78	711	2111	2451	59.45
80	685	1945	2246	59.99