ANTIOXIDANT IN BITUMEN

by

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CERTIFICATION OF APPROVAL

Antioxidant in Bitumen

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in partial fulfilment of the requirement for the

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(CIVIL ENGINEERING)

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CERTIFICATION OF ORIGINALITY

This is to certify that I am fully responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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AHMAD FAISAL BIN ABD. HALIM

ABSTRACT

Conventional virgin bitumen used in road pavement construction is susceptible to aging problem. Aging, or in its scientific term; oxidation reaction, occurs due to the presence of oxidizing agents (free radicals) that are formed during oxidation reaction in the bitumen. Oxidation in bitumen can cause an increase in asphalt viscosity as well as harden the bitumen, eventually loses its flexibility making it brittle and prone to cracking. This however can be counteracted by mixing the bitumen with antioxidant materials. In this project, the antioxidant material used is rice husk powder (75-micron size). The material contains lignin compound which could slows down the oxidation process via electron donation, preventing the free radicals from reacting with asphaltene (a vital compound in bitumen), and hence, prolong the lifespan of the bitumen. Not only the lignin-containing rice husk is environmental friendly, they are also cost-effective material since production price of rice husk powder is relatively cheaper than any other production of available bitumen modifier in the market. In this project, a total of three (3) types of modified bitumen have been prepared; virgin bitumen, 5%-containing rice husk bitumen, and 10%-containing rice husk bitumen. Two (2) laboratory aging tests which is Rolling Thin-Film Oven (RTFO) and Pressure Aging Vessel (PAV) tests have been conducted to simulate the aging of bitumen; short-term aging and long-term aging respectively. To support the evidence of antioxidizing properties of antioxidants towards virgin bitumen, a total of two (2) physical tests; Penetration Test and Softening Point Test have been conducted before and after the laboratory aging tests. From the laboratory tests results, the rice husk potential in term of improving current bitumen's service life are proven by analyzing the physical changes before and after the aging process of all samples. Up to this point, the rice husk bitumen possess better quality in term of lower rate of bitumen degradation (hardening effect), lesser amount of volatiles lost during short-term aging, and possess low temperature susceptibility value.

Keyword: *Bitumen; Aging; Oxidation; Antioxidant; Physical properties*

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CHAPTER 1

INTRODUCTION

1.1 Background Information

Bitumen, a naturally occurring mixture of polycyclic aromatic hydrocarbons, is a black, sticky, viscous material in pavement construction owing to its sticky nature to act as aggregates binder (Kanabar, A., 2010). According to the American Heritage Dictionary of the English Language, bitumen may exist naturally or obtained by distillation from coal or petroleum. Due to its unique features, bitumen is used worldwide in wide range of application, from road surfacing to waterproofing substance.

The promising global market of bitumen has forces the researchers worldwide to produce better and high-performance bitumen in order to secure a place in global market. Modified bitumen is approved to possess better performance compared to typical, conventional bitumen. Researchers have always studied the usual weakness of current virgin bitumen. This includes the susceptibility of bitumen towards aging problem caused by oxidation process. It has been noted that antioxidant materials (that contain lignin compound) may be the right answer to tackle bitumen aging. Thus, it is possible to invent a new breed of modified bitumen; Antioxidant-Containing Bitumen that are more resistive towards aging process.

It is also important to analyze the bitumen aging process, the factors lead to aging, several properties of bitumen that are susceptible to bitumen aging as well as finding the right way of combining antioxidant properties of lignin into virgin bitumen to produce a new breed of aging-resistive modified bitumen.

Therefore, extensive research on modified bitumen properties can be utterly beneficial in term of securing a place in the promising global bitumen market. Previous and on-going studies claim that current bitumen used for road and pavement are susceptible to **aging** problem. **Aging** (Oxidation) is caused by **external** surroundings (as shown in **Figure 1.1**) such as the presence of atmospheric oxygen, Ultra-violet radiation from the sun, temperature changes, traffic loadings, and the formation of free radicals (Kanabar, A., 2010)(Lu, X., 2008).

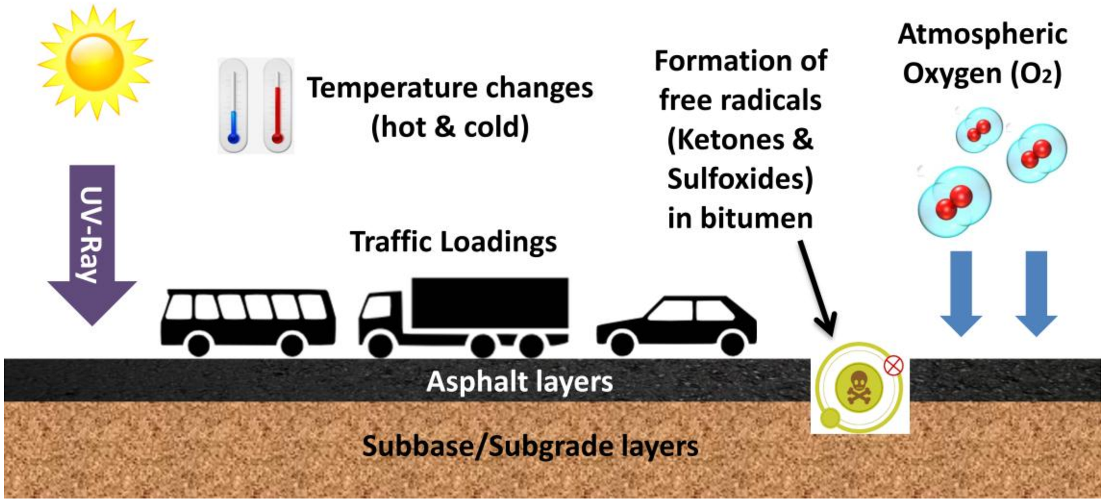

Figure 1.1: *Bitumen aging is caused by external surroundings*

In this project, the presence of oxidizing agents (free radicals) that are formed during oxidation reaction in the bitumen has been investigated and the reaction between lignin (antioxidant) compound and the free radicals (Ketones and Sulfoxides) in order to prevent bitumen aging has been studied. Theoretically, lignin can react which highly reactive free radicals in bitumen via electron donation, thus preventing the free radicals from reacting with asphaltene (a vital bitumen compound that determines the viscosity of bitumen) since oxidation in bitumen can cause an increase in asphalt viscosity as well as hardening the bitumen, eventually loses its flexibility to withstand traffic pressures as well as maintaining its uniform shape.

The bitumen eventually gets brittle and prone to cracking (**Figure 1.2**). Not only that, the current conventional pavement construction nowadays still cannot cope with other problems of pavement failure such as rutting (permanent deformation as shown in **Figure 1.3**), moisture damage or stripping failure (lack of adhesion and cohesion between bitumen binder and aggregates), and thermal cracking (when thermal stresses exceed the material's strength) (Kanabar, A., 2010).

Figure 1.2: *Typical longitudinal and transverse cracks*

Figure 1.3: *Severe ruts in an asphalt pavement*

In short, the conventional bitumen will eventually face aging problems that shorten its service life. **Figure 1.4** summarizes the concept of bitumen aging process that leads to road failure.

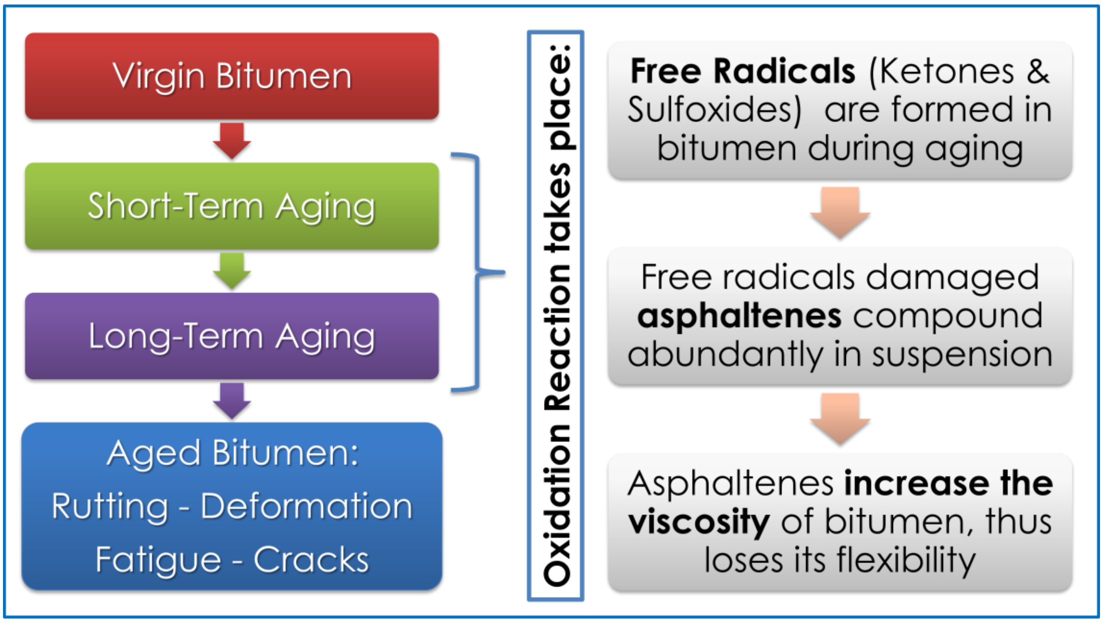

Figure 1.4: *Bitumen Aging Process*

Thus, it is utterly important to start a research to test for some of the best known antioxidant materials that are believed could anti-oxidize the free radicals (oxidizing agent) in bitumen. The said antioxidant contains **lignin** which is the driving agent that can slow down aging of bitumen via electron donation. As according to Williams R.C. (2008), via Fourier Transform Infrared (FTIR) spectroscopy testing, it is indicated that there are some antioxidant effects by the reduction in some of the various chemical aging products in the bitumen sample that contain lignin. Hence, it breeds the idea that any suitable lignin-containing products may benefits in term of retarding bitumen aging.

The early proposed antioxidant materials to be used for this research are rice husk, coconut shell, and palm shell as these materials contain lignin compound. But, it has been decided to only utilize **rice husk** (**Figure 1.5**) as the main antioxidant material for this project as this material is relatively easier to be prepared for laboratory testing compared to coconut shell and palm shell.

The project will only focus on the aspect of improving conventional virgin bitumen. Thus, the outcome of this project is to produce a new type of **modified bitumen** that can be used for Hot Mix Asphalt (HMA) production since the production of HMA requires Coarse Aggregate, Fine Aggregate, **Bitumen** and Filler (Portland cement) material. **Figure 1.6** illustrates the application of HMA in pavement construction.

Figure 1.5: *Suggested antioxidant material (rice husk)*

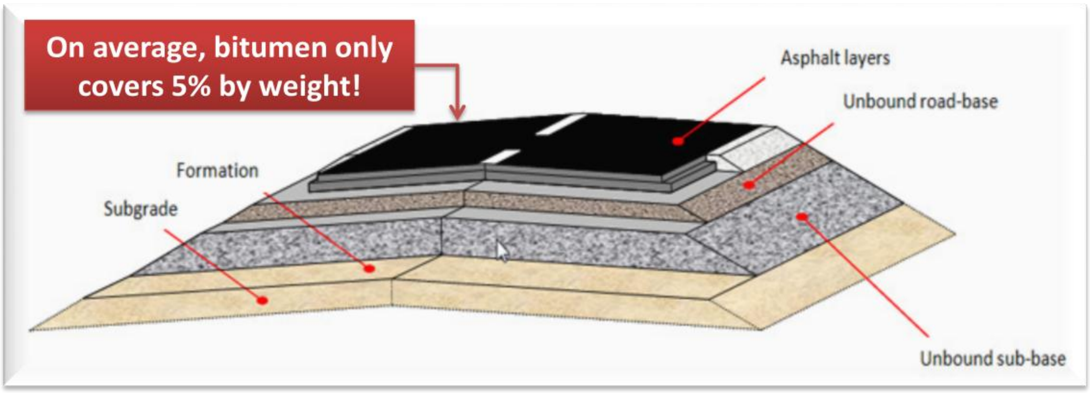

Figure 1.6: *Application of bitumen in HMA pavement construction*

In this project, an amount of rice husk has been grinded to get to its powdery form which can only pass 75 micron sieve filter. For this project, the performance of antioxidant as **filler material** in HMA production will not be tested as it will be more time-consuming and require several more laboratory tests that will be out of the scheduled FYP timeframe. Thus, the only focus of the project outcome is to produce a new type of cheap and environmental friendly **bitumen modifier** that could enhance current conventional bitumen; upon which the rice husk modified bitumen (RHB) can be made reliable to be used for Hot Mix Asphalt production.

At the end of this project, the performance of both bitumen containing rice husk and the virgin bitumen will be assessed. This objective can only be achieved by comparing the physical properties of both samples before and after laboratory aging process. The physical properties of bitumen are measured via Physical Test that includes Penetration and Softening Point Test. The laboratory aging process (Laboratory Aging Test) will be conducted by using **Rolling Thin-Film Oven** Test that simulates **short-term aging** (as similar to the condition during road construction period), and **Pressure Aging Vessel** (PAV) that simulates **long-term aging** (as similar as about 8-10 years of road service period).

1.2 Problem Statement

Throughout the years, aging of bitumen has always been the major factor of loss in efficiency of bitumen. As a result, the conventional HMA may possess a short service life and require regular maintenance in order to maintain its performance for another longer period (which is not cost-effective). Thus, researchers worldwide are driven to produce a reliable bitumen modifier that can be applied on conventional bitumen in order to disturb aging process and hence prolong the lifespan of bitumen. In this case, antioxidant material will be used as a bitumen modifier for our project.

Lignin-containing material like rice husk has always been disregarded as it has very limited potential in industry and is always seen as waste products. Typically, in Malaysia context, the cheapest way of dumping rice husk is by burning the material openly on the paddy field (right after harvesting process), as culturally, the burnt rice husk are believed to have the fertilizing properties that can nourish the growth of the to-be-planted paddy plants. This activity, however, contributes to air pollution Thus; this project of utilizing rice husk into bitumen modifier can change the industrial perception towards the full potential of rice husk. Not only that, this project is also an act of supporting the sustainable development in terms of utilizing waste materials for industrial benefits and preventing environmental pollution via open burning of rice husk.

Some previous studies have proven that antioxidant do have the potential to retard aging mechanism in bitumen. However, the issues of practicality and cost are some of the factors that make it unreliable. Therefore, this project, of utilizing cheap antioxidant material like rice husk powder could become another breakthrough in **Antioxidant/Bitumen** research. Most importantly, the proposed antioxidant material (rice husk) to be used is also an environmental friendly product that is abundantly available in Malaysia.

1.3 Objectives

The objectives of this research are:

- To understand **bitumen aging mechanism** (oxidation) in bitumen.
- To understand the **anti-oxidizing features of antioxidant** in preventing bitumen aging.
- To conduct a number of **laboratory tests** to test for the physical property changes of bitumen when added with rice husk powder.
- To **measure the degree of effectiveness** of the anti-oxidizing properties of rice-husk modified bitumen.

1.4 Scopes of Study

The scopes of study cover three major aspect; literature review, laboratory tests, and antioxidant's effectiveness:

- **Literature review**

i. The major property of bitumen that is prone to aging.
ii. Oxidation mechanism
ii. Antioxidant's (rice husk) anti-oxidizing properties.
iii. Previous researches on asphalt antioxidants.

- **Laboratory Tests**

i. Physical Test: Penetration & Softening Point Test conducted before & after laboratory aging process in order to investigate the physical changes that take place during laboratory aging process of all prepared bitumen samples. The physical hardness of all tested bitumen can be compared and the performance of each bitumen sample resisting aging can be determined at the end of the Physical Test after aging.

ii. Laboratory Aging Test: Rolling Thin-Film Oven (RTFO) & Pressure Aging Vessel (PAV) conducted to simulate short-term and long-term aging of the bitumen.

- **Antioxidant's Effectiveness**

i. Rate of hardening effects: How much percentage loss of bitumen degradation (rate of hardening effects) of the rice husk modified bitumen after laboratory aging process as compared to virgin bitumen.

ii. Penetration index & temperature susceptibility: Determined after the mixing process of bitumen with rice husk powder.

iii. Loss of volatiles: Measured after RTFO aging test.

1.5 Project Relevancy

This study is relevant in relation to the prospect of beneficial uses. Since global bitumen market is very promising nowadays; the need for innovating a more durable, flexible and stronger bitumen that are more resistive towards aging problem such as fatigue and rutting failure; are essential to cut short the operation and maintenance cost. The antioxidant in bitumen, if proven by laboratory tests to possess the potential to improve the current properties of virgin bitumen, will be a total breakthrough in the science of bitumen.

This project is also an act of promoting sustainable development in road/highway industry as we try to utilize waste materials (rice husk) into industrial benefits, in this case, prolonging bitumen lifespan, preventing open burning of rice husk, thus promoting preservation of environment in Malaysia.

This project is also relevant towards improving the previous researches regarding modified bitumen on which lack of promising laboratory results, impracticality of usage on bigger scale as well as cost factors. Since this project uses cheap products like rice husk and coconut shell as antioxidant, the results could be handy for further advance research to finally fortify the credibility of antioxidant in bitumen.

Not only that, this project may replace the current available bitumen modifier that usually uses chemical substance that is expensive and not environmental friendly. Hence, the application of rice husk, which is an environmental friendly product, has an added advantage over the current marketed bitumen modifier.

1.6 Project Feasibility (Within the Scope and Time Frame)

The proposed antioxidant (rice husk) material is very easy to prepare; only requires a 24-hours of oven heating (to remove moisture content), grinding of rice husk to 75-micron size powder, and simple mixing process using High Shear Mixer.

All of the required laboratory apparatus/equipments and machines are readily available in UTP Highway Laboratory.

Plus, all experiments conducted are not so time-consuming (guaranteed to be within Final Year Project semester (FYP1 and FYP2)).

CHAPTER 2

LITERATURE REVIEW

2.1 Conventional Bitumen versus Modified Bitumen

Due to population growth and economic development, extensive developments of asphalt-paved roadways are of high demand worldwide. As according to Transparency Market Research, in the website PR Newswire (2013), the global bitumen demand was 103.94 million tons in 2011 and is expected to reach 121.99 million tons in 2018. In terms of revenue, that is equivalent to USD 66.7 billion in 2011 and USD 84.42 billion in 2018. Meanwhile, Bodimeade, M. (2012) said, by 2017 the asphalt market is estimated to reach 126.3 million metric tons with the market growth predominantly driven by emerging markets of Asia-Pacific, Latin America, Middle East and Central & Eastern Europe. These staggering demand values has always been increasing year by year, and it calls for a more durable and more long-last asphalt pavement with low operation and maintenance cost.

Nowadays, STP Limited, a company based in Mumbai, India, has able to produce ShaliPMB, which is a Polymer Modified Bitumen, is produced by blending molten Bitumen with suitable thermoplastic polymers and additives (ShaliPMB, 2013). This achievement seems promising to penetrate the global bitumen market as STP Limited claimed that, the modified bitumen in general are more durable, has lesser susceptibility to temperature variations with superior performance under extremely cold or hot temperature conditions as well as able to prolong the bitumen lifespan by tackling aging and rutting problem as usually occurred in conventional bitumen.

In current era, with fast-paced development on track, the application of current virgin bitumen is not quite reliable in Malaysia (in term of maintenance cost). This particular issue has been raised by Jabatan Kerja Raya (JKR) Malaysia as reported by Utusan Malaysia (2011); the failure of asphalt roads constructed in Malaysia to meet the lifespan of minimum 10 years (before resurfacing required) is the major cause of increasing road maintenance cost annually. In the same article, Associate Professor Dr. M. Ratnasamy, Faculty of Engineering Universiti Putra Malaysia (UPM), has also addressed the long-term benefits of applying modified bitumen over conventional bitumen, such as the use of Polymer-Modified Bitumen technology that can helps the government of Malaysia to save about 20% of current road maintenance cost, equivalent to a RM100 million a year. Moreover, the handling (workability) of modified bitumen is also relatively easier as compared to conventional bitumen, further reducing the cost of excessive diesel usage and labor work force as the road construction period can be reduced significantly.

2.2 Bitumen Structure

Typical bitumen or crude oil contains aromatic hydrocarbon, asphaltene, resin and saturates (Mullins, et al, 2007). According to various scholars regarding binder structure, the major predominant molecule that determines the rheological properties of bitumen, in term of viscosity, is asphaltene (Luo, P. & Gu, Y., 2006) while an abundance of aromatics and saturates decrease the binder ductility (elastic effects) (Ruan, Y., 2003).

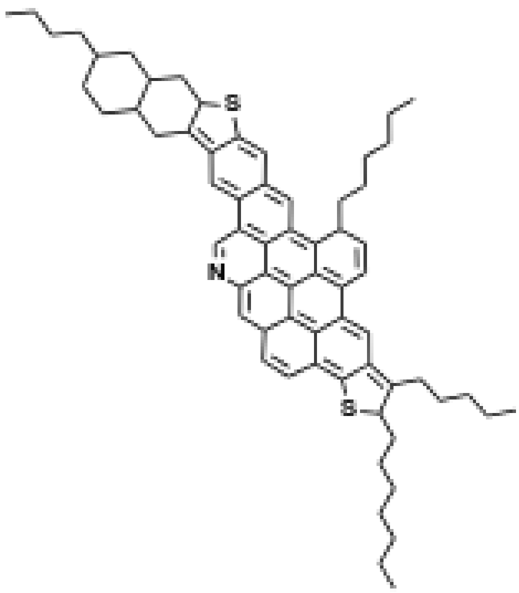

Figure 2.1: *Possible Asphaltene molecule*

2.3 Aging of Bitumen

One of the key factors to determine the lifespan of an asphalt pavement is by referring to the aging of bituminous binders. The process of aging involves chemical and physical property changes that usually make bituminous materials harder, more viscous and more brittle, thus increasing risk of pavement failure (Lu, X., 2008). Meanwhile, according to Mohamed, A.A. (2009), the durability of asphaltic concrete is greatly influenced by the environmental climate changes; hot and cold. High temperatures can soften the bitumen and consequently reduce the stiffness of asphaltic concrete, making the mix more susceptible to rutting. On the other hand, low temperature can increase the stiffness of bitumen and reduce the flexibility of the asphaltic concrete, causing fatigue failure as a result. These deteriorating features of the pavements are usually noted as aging of bitumen since the pavements are no longer exhibiting its great quality during its early lifespan.

Amit Kanabar (2010) suggested that the presence of oxygen in the atmosphere and ultraviolet radiation on its surface, along with changes in temperature, plays a significant role in the chemical aging process. The evidence of bituminous aging can be witnessed when the road starts to crack (longitudinal or transverse cracks) mainly due to thermal effects. Even worse, the cracks on the pavement surface may also increase aging of the bitumen due to increased exposure area to atmospheric oxygen. In short, the main mechanisms of bitumen aging are oxidation and loss of volatiles. As stated by Xiaohu Lu (2008), oxidative aging is an irreversible chemical reaction between components of bitumen and oxygen.

Generally, bitumen aging takes place in two stages; short-term aging and long-term aging (Kanabar, A., 2010). As according to Xiaohu Lu (2008), the first stage of bitumen aging usually occurred at high temperature during asphalt mixing, storage and construction process. During this stage, the binder is exposed to elevated temperatures and has larger contact area with the aggregates that can lead to rapid aging mechanism by volatilization and oxidation (Kanabar, A., 2010). Petersen (2006) termed this stage as “rapid spurt” reaction. Meanwhile, the long-term aging takes place at low (ambient) temperature during in-service on which the binder are surrounded with atmospheric oxygen that could percolate through the air voids of the binder and chemically reacts during the life of the in-service pavement (Kanabar, A., 2010). It is termed as long-term as it takes longer time than the short-term aging to react and form two primary oxidizing agents; noted as Ketone and Sulfoxide (Petersen, C. & Glaser, R., 2011).

2.4 Free Radicals Formation

Researchers have managed to short-list several free radicals chemical compounds that promote aging in bitumen such as Ketones and Sulphoxides.

According to Jeanie, L. D. (n.d.), free radicals are atoms or groups of atoms with an odd (unpaired) number of electrons and can be formed when oxygen interacts with certain molecules. Once formed these highly reactive radicals can start a chain reaction. In bitumen context, the free radicals, in nature, are in rampage to pair with another molecule, in this case, asphaltene.

It has been studied that Ketone formation has been identified as a major factor that leads to unstabilized asphaltene formation during oxidation process, and aggregation of asphaltenes have been shown to be the primary responsible for viscosity increase in bitumen (Petersen, C. & Glaser, R., 2011). This is also supported by Amit Kanabar (2010) who claimed that the ketones are formed at the first carbon of an alkyl chain attached to an aromatic ring, followed by the formation of sulfoxides from the oxidative aging reaction with the sulfur (exist primarily as sulfides) in bitumen.

As stated earlier, ketone is the major aging-product that could react with asphaltene. According to Peng Luo and Yongan Gu (2006), asphaltene can affect the viscosity of heavy crude oil as according to the existing suspension theories, the volume fraction and aggregation state of asphaltene particles are the main factors that increase the viscosity of heavy crude oil. Thus, if ketone production is not disturbed, the damaged asphaltenes will accumulates (in suspension) in bitumen, increasing its viscosity, making it inflexible and later on the bitumen will prone to cracking (fatigue) failure as the bitumen becomes more brittle.

2.5 Antioxidant Materials

In its simplest definition, an antioxidant is a molecule that inhibits the oxidation of other molecules. Antioxidants are molecules which can safely interact with free radicals (via electron donation) and terminate the chain reaction before vital molecules (asphaltenes) are damaged. This is as shown in **Figure 2.2**.

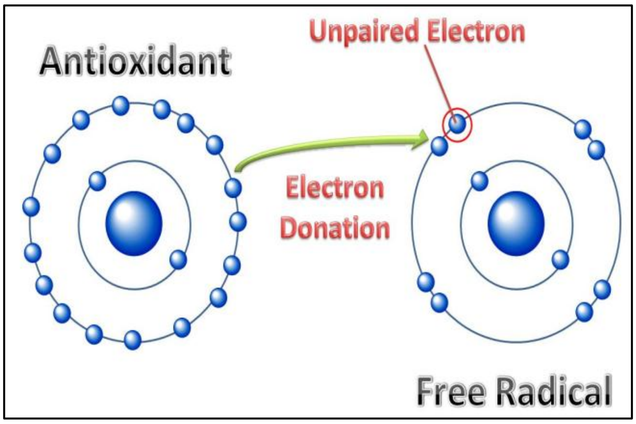

Figure 2.2: *How antioxidant works*

Since bitumen is susceptible to oxidation process, and that the process can reduce the service life of asphalt pavement, the antioxidant may in fact benefits bitumen in preventing aging mechanism. Previous studies have managed to identify a complex chemical compound that exhibits the major antioxidizing properties in most of antioxidant materials, which is **Lignin** compound.

2.5.1 Lignin

The lignin structure (Glazer, A.W. & Nikaido, H., 1995) is shown in **Figure 2.3**.

Previously (up to today), lignin-containing antioxidant material like rice husk is always disregarded as it has very limited potential in industry and is always seen as waste products of pulp and paper industry due to their very complex structure (Pouteau, C., 2003). Hence, the great challenge is to find new applications for lignin.

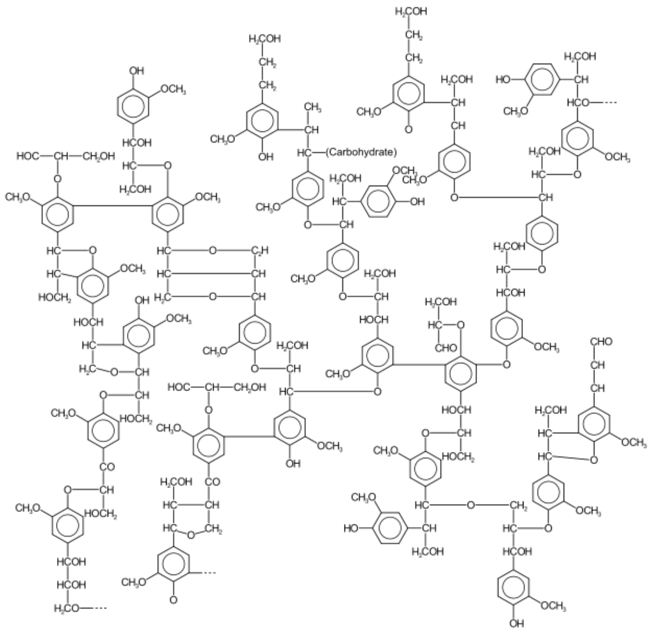

Figure 2.3: *Lignin Structure*

Fortunately, lignin is able to react with free radicals in term of donating its electron in order to stabilize the highly reactive free radicals (Ketones and Sulfoxides). Thus, the asphaltene compound in bitumen will remain undisturbed and theoretically, the bitumen possesses longer lifespan as compared to the one that does not contain antioxidant.

2.5.2 Rice Husk

According to Habeeb, G. A. (2010), of all the 649.7 million tons of rice produced annually worldwide, 20% of the portion is the residual of the agricultural product called rice husk. Rise husk (or rice hull) is the hard protecting coverings of rice grain from paddy plant. Traditionally, rice husk has always either being burnt or dumped as waste products. Meanwhile, the utilization of rice husk as fuels for instance, can contribute to air pollution.

Thus, researchers have now managed to change the stereotype function of rice husk into several valuable and more eco-friendly materials for building construction and insulation purpose. For example, Rice Husk Ash (RHA) has been used for the production of flat steel (plate product or a hot rolled strip product) that is typically used for automotive body panels (Bronzeoak Ltd, 2003). Not only that, RHA has also been optimized for the manufacturing of low cost building blocks and in the production of high quality cement (Bronzeoak Ltd, 2003).

The bright future of rice husk has now been taken into advantage for the project; which is to produce a bitumen modifier made solely from rice husk. This material is very suitable to be used for the project as according to Olivier, P. A. (2004), rice husk's chemical component consist of approximately 20% opaline silica in combination with a large amount of lignin (phenyl propanoid structural polymer).

2.6 Previous Studies (Asphalt Antioxidants)

Ouyang, et al. (2005) has managed to investigate the effects of antioxidant in Polymer-Modified Asphalt (PMA), and the result was positively successful. Their research team has added zinc dialkyldithiophosphate (ZDDP) and zinc dibutyl dithiocarbamate (ZDBC) to PMA upon which the modifiers are approved to possess antioxidant ability. The PMA plus modifiers (ZDDP and ZDBC) are more resistant to the formation of carbonyl, indicating the improvement of aging resistance of the binder. Moreover, the ZDDP and ZDBC as antioxidants could retard the oxidation of the PMA through the inhibition of peroxides and radical scavenging.

Meanwhile, McCready, N.S. and Williams, R.C. (2007) had also managed to test the effects of antioxidant (wood lignin) towards bitumen aging mechanism. They have utilized agriculturally derived lignin as an antioxidant in bitumen. According to them, the addition of lignin to bitumen can cause a slight stiffening effect depending upon the percentage and type of lignin used. At high lignin contents, the asphalt's grade gradually increase at high temperature, while at low temperature, the effects were slightly negative but of no significant at all.

CHAPTER 3

METHODOLOGY

3.1 Antioxidant / Bitumen Preparation

The antioxidant material (rice husk powder) has been successfully prepared for the project and the modifier has been mixed with virgin bitumen in an appropriate amount of rice husk powder (5% and 10%). Shown below are the rice husk powder production steps as well as the steps that are taken during the antioxidant/bitumen mixing process:-

3.1.1 Antioxidant Material Preparation

1. An amount of rise husk (in its original form) has been dried in the drying oven for 24 hours at 100°C to get rid of any moisture content in it.
2. The dried rice husk is then being grinded using grinding machine (**Figure 3.1**) until it gets to powder form.
3. The powdered rice husk has been sieved using 75 micron sieve machine. Only the fine particles that passed the 75 micron sieve are collected (**Figure 3.2**) for the next step (mixing with virgin 80/100 grade bitumen).

Figure 3.1: *Grinding Machine*

Figure 3.2: *Sample from the grinded 75-micron rice husk powder*

3.1.2 Antioxidant/Bitumen Mixing Process:

1. A three (3) sets of 1-Litre empty can were weighed and recorded. Every can were then labeled "*0% Rice Husk*" for Can #1, "*5% Rice Husk*" for Can #2, and "*10% Rice Husk*" for Can #3.

2. An amount of virgin bitumen was poured in each of the can after being heated until reaching pouring state. After the bitumen has cooled down to room temperature, each can were weighed and recorded accordingly.

3. By using below equation, the weight of rice husk required for Can 2 and Can 3 to be mixed with the virgin bitumen can be determined:

   <u>For 5% rice husk content:</u>

   Rice Husk Weight (g) $= \frac{5\%}{95\%}$ X (B - A)

   where, A = Weight of Empty Can (in gram)

   B = Weight of Empty Can + Bitumen (in gram)

   <u>For 10% rice husk content:</u>

   Rice Husk Weight (g) $= \frac{10\%}{90\%}$ X (B - A)

4. After determining the rice husk weight required for Can #2 and Can #3, amounts of rice husk were weighed to the desired weight.

5. Can#2 were then placed on the hotplate heater machine set to an appropriate temperature sufficient enough to keep the bitumen in pouring state throughout the mixing process.

6. High Shear Mixer machine stirrer was placed into the bitumen as shown in **Figure 3.3** and the previously weighed rice husk powder for Can#2 (that would contain 5% rice husk powder) were slowly inserted into the can at the same moment when the stirrer were allowed to thoroughly mix the rice husk powder with the bitumen until both ingredients were uniformly mixed.

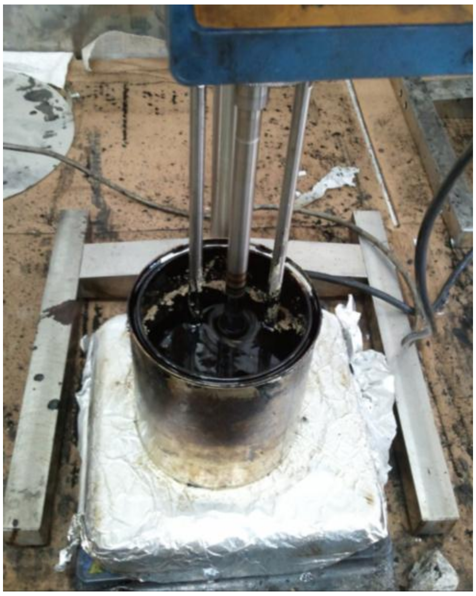

Figure 3.3: *Mixing process of virgin bitumen with rice husk powder*

7. The procedure is then repeated to Can#3 that would contain 10% rice husk powder.
8. Can#1 was not being mixed with any rice husk powder since it was designated as control sample (virgin bitumen).

3.2 Physical Test (Before Aging)

The main objective of conducting Physical Test before laboratory aging test is to measure the initial physical properties of the bitumen samples that contain rice husk (5% and 10%), and the one without rice husk content (virgin bitumen). **Penetration Test** and **Softening Point Test** have been conducted to measure the penetration and softening point of all bitumen samples. Basically, these two properties are correlated to each other in terms of hardness and temperature susceptibility of the samples. Penetration Index graph can also be calculated and plotted by integrating both penetration and softening point data into a single graph.

The effectiveness of anti-oxidizing properties of rice husk powder towards the bitumen in term of preventing aging can also be assessed. By comparing the data from Physical Test before and after aging, the degree of bitumen degradation can be obtained. The percentage difference in degradation values between virgin bitumen and rice husk bitumen will be determined at the end of this project. Hypothetically, the rice husk bitumen should age insignificantly as compared to the virgin bitumen.

This can be measured in terms of how much reduction in penetration value and increment of softening point value that all samples undergo after laboratory aging process, indicating the degree of hardness increases after the aging process.

By right, the samples containing rice husk should not harden to a much more degree than the virgin bitumen as oxidation reaction has been prevented due to the anti-oxidation reaction posed by the rice husk powder (lignin compound) towards the bitumen.

3.2.1 Penetration Test

According to BP Bitumen Australia website, Penetration Test can be applied on bitumen to determine the consistency of bitumen by measuring the distance that a standard needle will penetrate vertically into a sample (reported in tenths of a millimeter) under specified conditions of:-

- Loading = 100g,
- Temperature = 25°C, and
- Time = 5 seconds

Semi Automatic Penetrometer was adopted for this test (**Figure 3.4**). The standard procedure of this method is in AASHTO T 49 and ASTM D 5: Penetration of Bituminous Materials (*Pavement Interative*).

3.2.1.1 <u>Basic Procedure of Penetration Test</u>

1. The virgin bitumen sample was heated until it becomes fluid.
2. The heated bitumen sample was placed in a penetration cone such that when cooled, the depth of the sample is 10 mm greater than the expected penetration. For this experiment, 3 sets of cones containing bitumen sample were prepared.
3. The bitumen temperature is cooled in water bath so that its temperature maintained at 25°C.

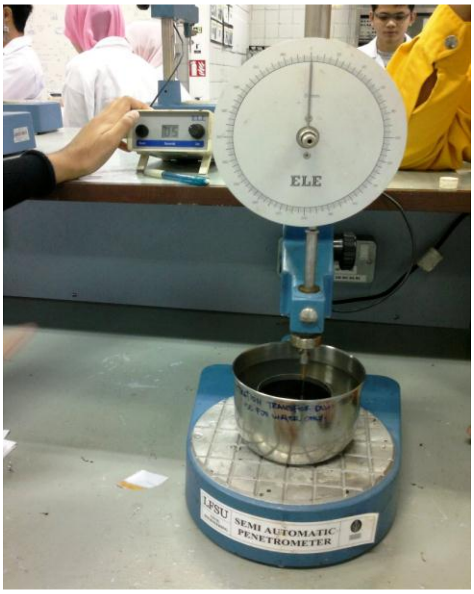

Figure 3.4: *Semi-Automatic Penetrometer*

4. The cooled bitumen was then placed below the needle, such that the needle just touches the bitumen surface.

5. Before starting the penetration, the load is set at 100g. The needle was then allowed to penetrate the bitumen and the test was stopped after 5 seconds.

6. The results were recorded in decimillimetre on which one penetration equals 1 dcm or equivalent to 0.1 mm.

7. At least three (3) readings per cone were taken in order to confirm the accuracy of the result. The next needle point must not less than 10mm from the side of the container and not less than 10mm apart from previous points.

8. After three penetration values for every cup has been determined for all of the three cups, the test was repeated to bitumen samples containing rice husk (5% and 10%).

3.2.2 Softening Point Test

According to *Pavement Interactive* (2007) website, the softening point is defined as the temperature at which a bitumen sample can no longer support the weight of a 3.5-g steel ball. Basically, two horizontal disks of bitumen, cast in shouldered brass rings (**Figure 3.5**), are heated at a controlled rate in a liquid bath (**Figure 3.6**) while each supports a steel ball. The softening point is reported as the mean of the temperatures at which the two disks soften enough to allow each ball, enveloped in bitumen, to fall a distance of 25 mm (**Figure 3.7**).

The standard procedure of this test can be viewed in AASHTO T 53 and ASTM D 36: Softening Point of Bitumen (Ring-and-Ball Apparatus).

Figure 3.5: *Shouldered brass ring and ball*

3.2.2.1 Basic Procedure of Softening Point Test

1. The virgin bitumen sample was heated between 75 ^{o}C and 100^{o}C until it reached to pouring state. The sample was then stirred to remove air bubbles.
2. A set of brass rings were heated and glycerine was applied beneath the ring. The sample was filled into the ring and allowed to cool for 30 minutes.
3. Excess sample were removed with the help of a warmed, sharp knife.
4. The apparatus with the rings (containing sample), thermometer and ball guides were assembled in position and placed inside a beaker that is filled with distilled water cooled down to 5.0 ^{o}C ± 0.5^{o}C using ice cubes. The apparatus was left for 15 minutes with the temperature maintained.
6. With the help of magnetic stirrer, the liquid was stirred and heat was applied to the beaker to increase the temperature of water at a uniform temperature rate of 5.0 ± 0.5^{o}C per minute.

7. The heat was applied until the sample softens and the ball was allowed to pass through the ring.

8. The temperature at which the ball touches the bottom plate was recorded as the softening point temperature of the sample.

9. The test was repeated again for bitumen samples that contain 5% rice husk and 10% rice husk.

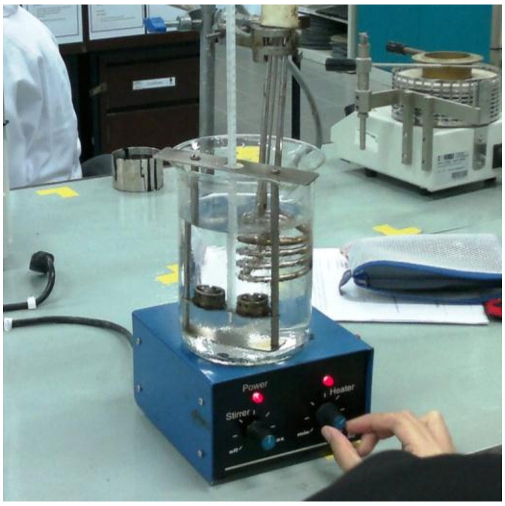

Figure 3.6: *Controlling water bath temperature at uniform rate (5.0 ± 0.5 ^{o}C per minute)*

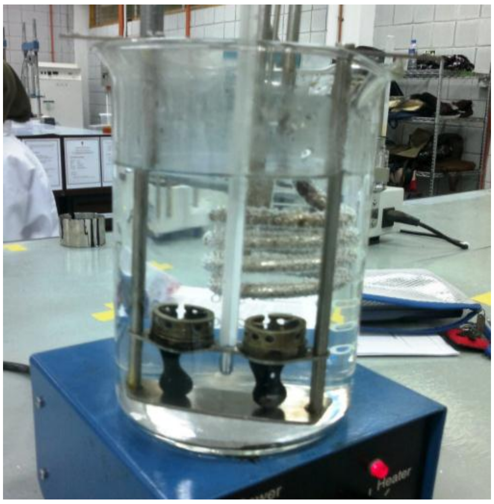

Figure 3.7: *Bitumen soften, letting the ball to fall at softening point temperature*

3.3 Laboratory Aging Tests

To test for asphalt performance pertaining to aging resistivity after mixing with antioxidant, there are two major laboratory tests that previous researchers usually apply; Rolling Thin-Film Oven (RTFO) Test and Pressure Aging Vessel (PAV) Test. These two tests are preferred as RTFO can simulate the short-term aging while PAV can simulate the long-term aging (Kanabar, A., 2010).

3.3.1 Rolling Thin-Film Oven (RTFO) Test

This laboratory test is very significant in providing the best simulation of short-term aging as usually occurred during hot mixing and the placement process (Kanabar, A., 2010). As stated in *Pavement Interactive* (2011) website, The RTFO test also provides a quantitative measure of the volatiles lost during the aging process. The basic RTFO procedure takes unaged bitumen samples in cylindrical glass bottles and then places these bottles in a rotating carriage within an oven. At high temperature of 163°C, the carriage will rotate at 15 RPM within the oven and ages the samples for 85 minutes. The rotation effects are significant as it continuously exposes the bitumen with heat and air flow as well as slowly mixes each sample. Samples are then stored for use in physical properties tests or to be used for PAV test.

Figure 3.8: *RTFO Test Machine*

The standard RTFO test procedure can be found in AASHTO T 240 and ASTM D 2872 "Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)".

3.3.1.1 Basic Procedure of RTFO Test

1. A sample of bitumen (virgin) was heated until it is fluid to pour. The sample was then stirred to ensure homogeneity and to remove air bubbles.

2. An empty RTFO bottle was weighed and recorded as W_o. This is designated as the "mass change" bottle.

3. 35g of bitumen sample are poured into the bottle (**Figure 3.9**) and immediately the bottles are turned on their side without rotating or twisting and placed on a cooling rack. Step 1 and Step 2 are repeated for other bitumen samples containing different amount of antioxidant (5% and 10%).

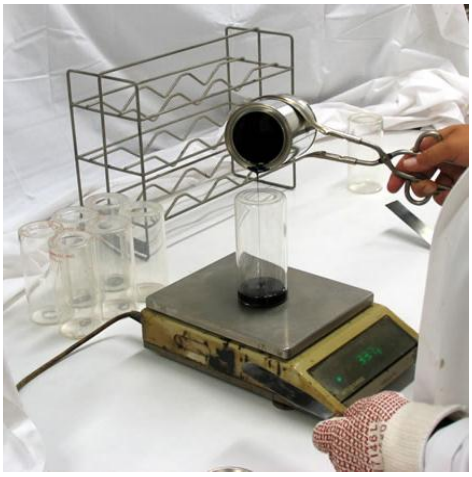

Figure 3.9: *Pouring bitumen sample to each bottle*

4. All bottles were allowed to cool for 60 to 180 minutes.
5. After cooling, each bottle was weighed again and recorded as W_{bi}.
6. The bottles were then placed in the RTFO oven carousel, and after closing the door, the carousel was rotated at 15 RPM for 85 minutes. During this time, the oven temperature was maintained at 163°C and the airflow into the bottles was maintained at 4000 mL/min.
7. The bottles were removed one at a time from the carousel. The residues from each bottle were removed by first pouring as much material as possible, then the sides of the bottle was scraped to remove any remaining residue. There is no standard scraping utensil but at least 90% of the bitumen should be removed from the bottle. RTFO residue should be tested within 72 hours of aging.
8. Each bottle was then weighed and recorded as W_{bf}.
9. The procedures were repeated for the bitumen samples that contain 5% and 10% rice husk content.

3.3.1.2 Basic Analysis of RTFO Test

After the aged bitumen sample has been collected, the calculation of the mass change of bitumen sample can be performed as below (*Pavement Interactive*, 2011):-

$$\textbf{Mass Change} = \frac{(\mathbf{A} - \mathbf{B})}{\mathbf{A}} \ (\mathbf{100})$$

where $A = W_{bi} - W_o$ $B = W_{bf} - W_o$

and W_{bi} = bottle + bitumen weight before aging

W_{bf} = bottle + bitumen weight after aging

W_o = empty bottle weight

A = initial sample weight

B = final sample weight

The loss in mass of the sample will indicate the mass quantity of volatiles lost from the bitumen during short-term aging.

3.3.2 Pressure Aging Vessel (PAV) Test

In order to simulate the long-term aging of bitumen, Pressure Aging Vessel (PAV) test is most preferred by researchers. As stated earlier, long-term aging occurred during in-service of asphalt pavement where the effects of thermal and load-induced loads gradually crack the pavement, causing fatigue failure (Kanabar, A., 2011). The PAV test can age the bitumen sample that simulates the in-service aging over 7 to 10 year period. The bitumen sample will be exposed to heat and pressure during PAV test and the simulated long-term aged bitumen sample can be used for physical property testing (*Pavement Interactive*, 2011).

Figure 3.10: *Pressure Aging Vessel (PAV)*

The basic PAV procedure takes RTFO aged bitumen samples and exposes them to high air pressure up to 305 psi (2.10 MPa or 20.7 atm) and heated for 20 hours. The heating temperature, as according to Amit Kanabar (2011), can vary according to different climate simulation. The proposed heating temperature that simulates different climate is as below:-

Temperature	Simulation
90°C	Cold climate
100°C	Moderate Climate
110°C	Hot Climate

For application in Malaysian climate, the preferred simulation is Moderate climate at which the PAV heating temperature test will be set at 100°C. The standard PAV procedure is embodied in AASHTO R 28: "Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)"

3.3.2.1 Basic Procedure of PAV Test

1. The RTFO-aged virgin bitumen samples were heated until fluid enough to pour. The samples were then stirred and poured (50 g) into a preheated thin

film oven pan (**Figure 3.11**). This step is repeated for other bitumen sample containing antioxidant.

Figure 3.11: *Preheated thin film oven pan*

Figure 3.12: *Pan Holder*

2. All pans were placed in a pan holder (**Figure 3.12**) and placed inside preheated PAV (**Figure 3.13**).
3. PAV machine was sealed and the temperature was set at 100°C.
4. Once the PAV has reached the desired temperature, the PAV was then pressurized to 300 psi (2.07 MPa) for 20 hours.
5. At the end of the aging period, the pressure was gradually released and the pans were removed from the PAV.
6. The container (containing all samples) was then placed in a vacuum oven (**Figure 3.14**) at 170°C and the samples were degassed for 30 minutes to remove entrapped air. If not degassed, entrapped air bubbles may cause premature breaking in the DTT test.
7. After removing the sample from vacuum oven, the sample is now ready to be used for conducting Physical Test (after aging).

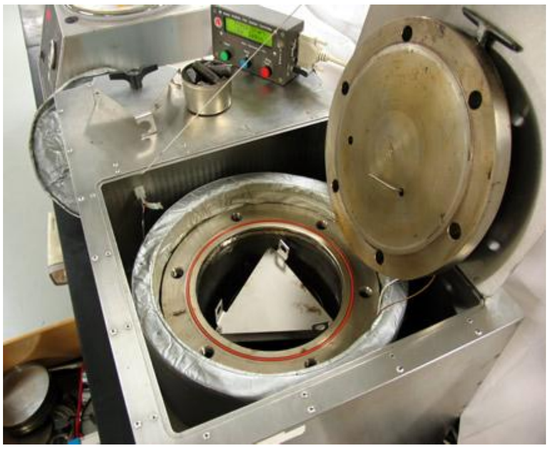

Figure 3.13: *Pan Holder inside preheated PAV*

Figure 3.14: *Vacuum oven*

3.4 Physical Test (After Aging)

After all bitumen samples have been aged, the next step would be to test for their physical properties in order to access the effectiveness of aging-resistivity potential of bitumen that has been mixed with antioxidant. Only by these test, the true verdict of reliability of antioxidant in bitumen can be testified. At the end of the Physical Test after aging, the results of physical properties of all tested bitumen samples before and after the aging process have been compared and analyzed.

The proposed physicals tests for this research are **Penetration Test** and **Softening Point Test** and the procedures of these experiments followed the same procedures as listed in section **3.2: Physical Test (Before Aging)**.

3.5 Penetration Index

Penetration Index (PI) represents a quantitative measure of the susceptibility of bitumen towards variation in temperature. Penetration index is very important so as to predict the behavior of the bitumen for any application, in this case, the behavior of bitumen during HMA production as well as during in-service period. PI provides a more reliable information regarding the bitumen grade as it integrates both the Penetration value and Softening Point value into a single equation. It determines the degree of susceptibility of bitumen towards temperature changes, on which the lower the PI value, the more susceptible the bitumen towards temperature changes.

Several equations exist that define the way that the viscosity (or consistency) changes with temperature. One of the best known is the one developed by Pfeiffer and Van Doormaal (Ehinola et al, 2012) which states that: If the logarithm of penetration, *P*, is plotted against temperature, *T*, a straight line is obtained such that:

$$\log P = AT + K \qquad (1)$$

where

A = The temperature susceptibility

P = Penetration at temperature T

K = Constant

Pfeiffer and Van Doormaal developed an equation for the temperature response that assumes a value of about zero for road bitumen. For this reason they defined the penetration index (*PI*) as:

$$\frac{20-PI}{10+PI} = 50A \qquad (2)$$

or explicitly,

$$PI = \frac{20-500A}{1+50A} \qquad (3)$$

For Grade 80/100 bitumen, as according to ORJ Group Website, the typical range of PI value is from -0.8 to +0.7. The *PI* is an unequivocal function of *A* and hence it may be used for the same purpose. The values of *A* and *PI* can be derived from penetration measurements at two temperatures, *T1* and *T2* using the equation:

$$A = \frac{\log Pen1 - \log Pen2}{T1 - T2} \qquad (4)$$

where,

Pen_1 = Penetration at temperature (T_1) of 25°C.

Pen_2 = Penetration at softening point temperature (T_2)

Meanwhile, the consistency of bitumen samples at the softening point can be expressed in terms of linear extrapolation of logarithm of penetration (Pen1) versus softening point temperature (T2).

Pfeiffer and Van Doormaal (Read, J., 2013) found that most bitumen had a penetration of about 800 dmm (deci-millimetre) at the ASTM softening point temperature. By replacing *T*2 in the above equation by the ASTM softening point temperature and the penetration at *T*2 by 800 the equation can now be simplified as:

$$A = \frac{\log Pen1 - \log 800}{25°C - T2} \qquad (5)$$

In this project, equations (3) and (5) will be applied to calculate first for *A-value* (temperature susceptibility of bitumen) and *PI* (penetration index). These were calculated from the measured softening point temperatures and penetrations of every bitumen sample (virgin, 5% rice husk, and 10% rice husk).

3.6 Significance of Laboratory Test Results

For the sake of proving the hypothesis that antioxidant can give positive impact towards bitumen, especially in tackling aging problem, all of the laboratory tests are utterly important since measurement data can be analyzed and concluded to a concrete statement. The significance of all laboratory tests conducted is as listed:-

Rolling Thin-Film Oven (RTFO) Test	• The bitumen sample can be short-term aged to simulate the hot mixing as well as during pavement laying. • The lost of volatiles can also be determined by calculating the mass change after the aging test.
Pressure Aging Vessel (PAV) Test	• The test can long-term aged the RTFO aged sample to simulate the in-service life of over 7 to 10 years period. • After the test, the bitumen sample can considered fully aged as it has covered short-term aging as well as long-term aging.
Penetration Test	• The consistency of aged bitumen can be determined. If the bitumen containing antioxidant is more resistive towards

	aging, the penetration grade of the bitumen should not degrade significantly. • The effectiveness of each type of antioxidant can be compared and the best to lowest effective antioxidant can be identified.
Softening Point Test	• This test indicates the hardness of bitumen. If the softening point of aged bitumen is higher than the unaged bitumen, the bitumen is considered to be increasing in hardness. By this means, if the bitumen containing antioxidant cannot reduce the effect of hardening after the aging process, the antioxidant is considered as failed to meet the objective.

3.6 Gantt Chart

In order to ensure that the direction of this project leads to success, a Gantt chart has been prepared to advance the key milestones as well as to monitor the project flow throughout the Final Year Project (FYP1 and FYP2) semester. The Gantt chart can be viewed **Table 3.1** (FYP1) and **Table 3.2** (FYP2).

Table 3.1: *Timeline for FYP1*

No	**Detail/Week**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**MID SEMESTER BREAK**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1.	Selection of Project Topic	■														
2.	Finalization on Project Topic with Assigned Supervisor		■													
3.	Literature Review			■	■	■										
4.	Submission of Draft Extended Proposal					■										
5.	Submission of Extended Proposal						●									
6.	Preparation on Proposal Defense							■		■						
7.	Proposal Defense									■	●					
8.	Continuation of Project Works											■	■	■		
9.	Submission of Interim Draft Report														●	
10.	Submission on Interim Report															●

Table 3.2: Timeline for FYP2

No.	Detail/Week	1	2	3	4	5	6	7	Mid-Semester Break	8	9	10	11	12	13	14	15
1	**Laboratory Samples Preparation**																
	• Highway Laboratory Safety Briefing	■															
	• Antioxidant / Bitumen Mixing	■	■	■	■												
2	**Laboratory Tests**																
	• Physical Test 1 (Before Aging)			■	■	■											
	• Aging Test									■	■	■					
	• Physical Test 2 (After Aging)											■	■	■			
3	**Data Collection & Compilation**			■	■	■	■	■		■	■	■	■	■			
4	**Progress Report Preparation**																
	• Submission of Draft Report							■									
	• Submission of Progress Report									●							
6	**Pre-SEDEX**												●				
7	**Project Dissertation Preparation**																
	• Submission of Draft Report													●			
	• Submission of Project Dissertation (Soft bound)														●		
	• Submission of Project Dissertation (Hard Bound)																●
8	**Submission of Technical Paper**														●		
9	**Oral Presentation**															●	

● Key Milestone ■ Process

CHAPTER 4

RESULTS & CALCULATIONS

Up to this stage, all results of the **Physical Tests** which is Penetration Test and Softening Point Test before and after the aging process have been made available for further discussion. In this project, three samples were analyzed which is the:

a) **virgin** 80/100 grade bitumen that does not contain any antioxidant,
b) 80/100 grade bitumen that contains **5%** rice husk powder, and
c) 80/100 grade bitumen that contains **10%** rice husk powder.

4.1 Penetration Test (Before Aging) Results

For this test, three penetration cones have been prepared for every sample (virgin, 5%, and 10%) with three determinations per cone in order to improve the consistency of the results. The results of the penetration grade for the three samples (virgin, 5%, and 10%) before the aging process were as shown in **APPENDIX 1**. The graph showing the relationship between different amounts of rice husk powder content with the bitumen penetration grade is as shown in **Figure 4.1** below.

4.2 Softening Point Test (Before Aging) Results

The softening point of every sample (virgin, 5%, and 10%) have been successfully obtained. Note that 3 sets of experiments have been conducted for each of the sample in order to maximize the results' consistency as well as eliminating data that were out of range. The results were recorded as in **APPENDIX 2** and the graph showing the effects of adding different amount of rice husk powder is as shown in **Figure 4.2**.

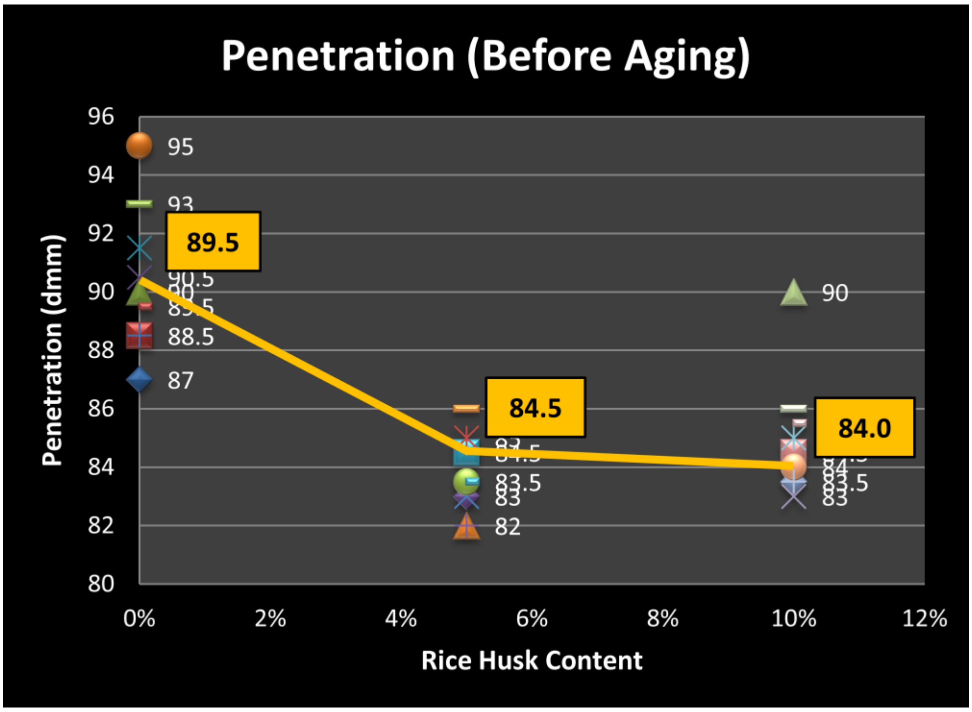

Figure 4.1: *Penetration (Before Aging) versus addition of rice husk*

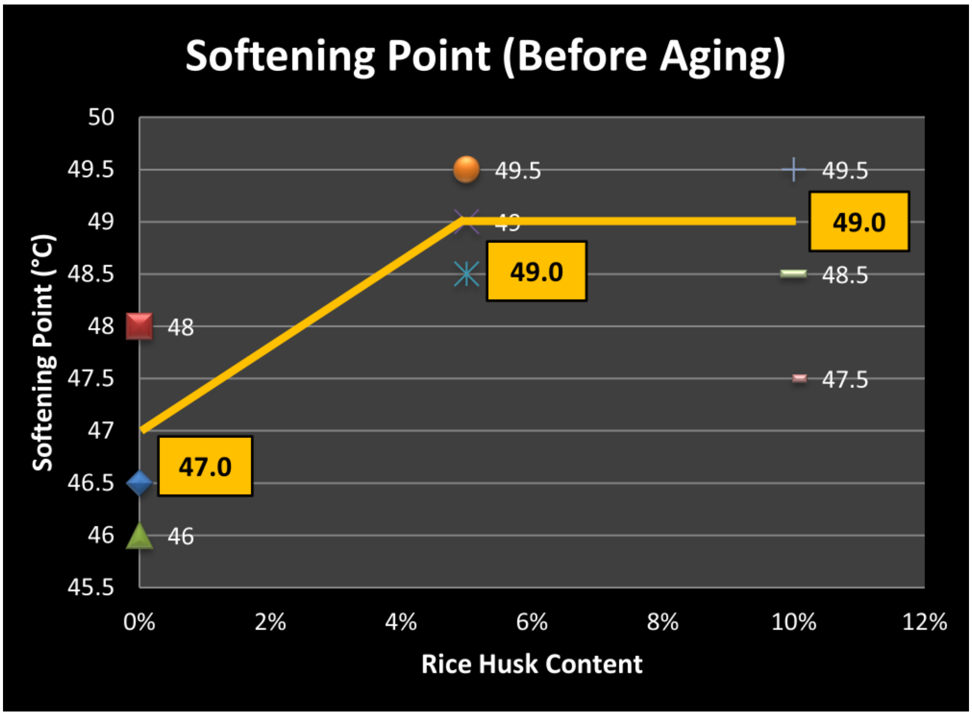

Figure 4.2: *Softening Point (Before Aging) versus addition of rice husk*

4.3 RTFO Mass Change Calculation

As stated in section **3.3.1.2: Basic Analysis of RTFO Test**, the mass change of bitumen samples before and after aging test has been calculated and as shown in **Table 4.1** and **Figure 4.3**.

Table 4.1: *Average Mass Quantity of Volatiles Lost After Aging*

Rice Husk Content	Wbi (gram)	Wbf (gram)	A (gram)	B (gram)	A - B (gram)	[(A-B)/A] X 100 (%)	Average losses (%)
0%	207	203	37	33	4	10.8	7.2
	206	204	36	34	2	5.6	
	208	206	38	36	2	5.3	
5%	206	204	36	34	2	5.6	6.4
	207	205	37	35	2	5.4	
	207	204	37	34	3	8.1	
10%	206	204	36	34	2	5.6	5.6
	204	202	34	32	2	5.9	
	208	206	38	36	2	5.3	

Note:-

Empty bottle weight, Wo = *170 gram*

Wbi = *Empty bottle + Bitumen before aging*

Wbf = *Empty bottle + Bitumen after aging*

A = *Wbi - Wo*

B = *Wbf - Wo*

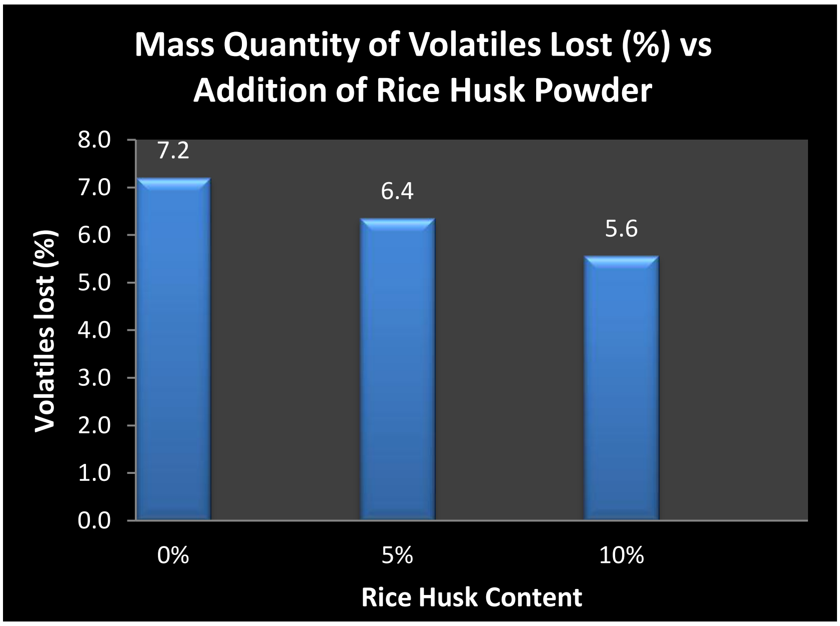

Figure 4.3: *Average mass quantity of Volatiles lost after aging*

4.4 Temperature Susceptibility & Penetration Index Computation

After obtaining the results of Penetration and Softening Point before aging process, the Penetration Index can now be computed using equations as stated before in section **3.4: Penetration Index**. The results of Temperature Susceptibility (A-value) and Penetration Index of bitumen samples with different amount rice husk content are as shown in **Table 4.2**, **Figure 4.4** and **Figure 4.5**. Note that, only average data of penetration and softening point are considered.

Table 4.2: *Penetration Index (Before Aging) Results*

Samples	Penetration Grade		Softening Point		Temperature Susceptibility	Penetration Index
	P1	P2	T1	T2	A	PI
Virgin Bitumen	89.5	800	25	47	0.0432	-0.5123
Bitumen with 5% Rice Husk Content	83	800	25	49	0.0410	-0.1641
Bitumen with 10% Rice Husk Content	85	800	25	48.5	0.0414	-0.2333

Note: 1) *P1 = Penetration at temperature T1, 25°C (ASTM standard temperature for penetration test)*
2) P2 = Penetration at temperature T2 (the softening point temperature). Most bitumen's penetration is 800 dmm at its softening point temperature.
3) A = Ratio of log penetration with temperature change [Equation (5)]
4) Penetration Index value [Equation (3)] is typically in a range of -0.8 to +0.7 for grade 80/100 bitumen.

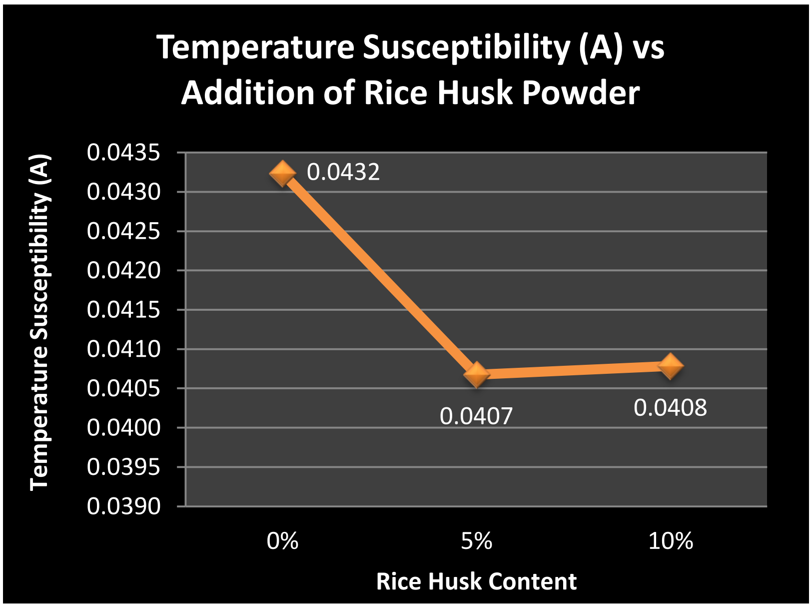

Figure 4.4: *Temperature susceptibility versus addition of rice husk*

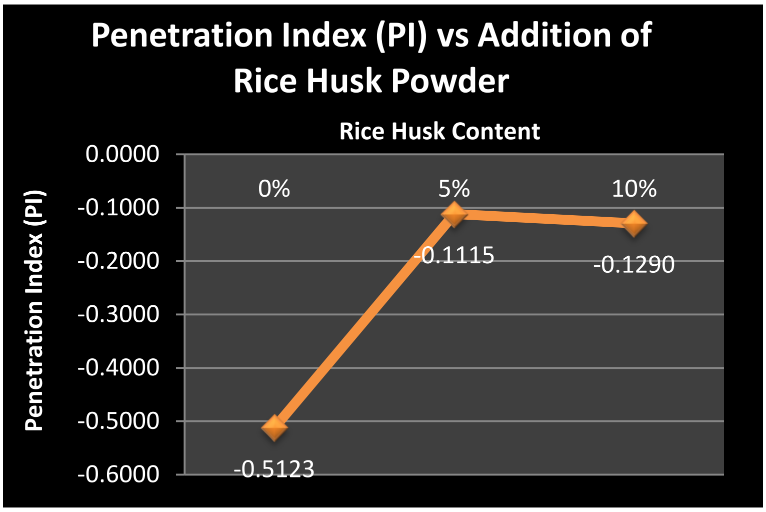

Figure 4.5: *Penetration Index versus addition of rice husk*

4.5 Penetration Test (After Aging) Results

The results of the penetration grade for the three samples (virgin, 5%, and 10%) after the aging process were as shown in **APPENDIX 3**. The graph showing the relationship between different amounts of rice husk powder content with the bitumen penetration grade is as shown in **Figure 4.6**. Only two penetration cones (instead of three, due to time constraint) have been prepared for every sample (virgin, 5%, and 10%) with three determinations per cone in order to minimize percentage of errors.

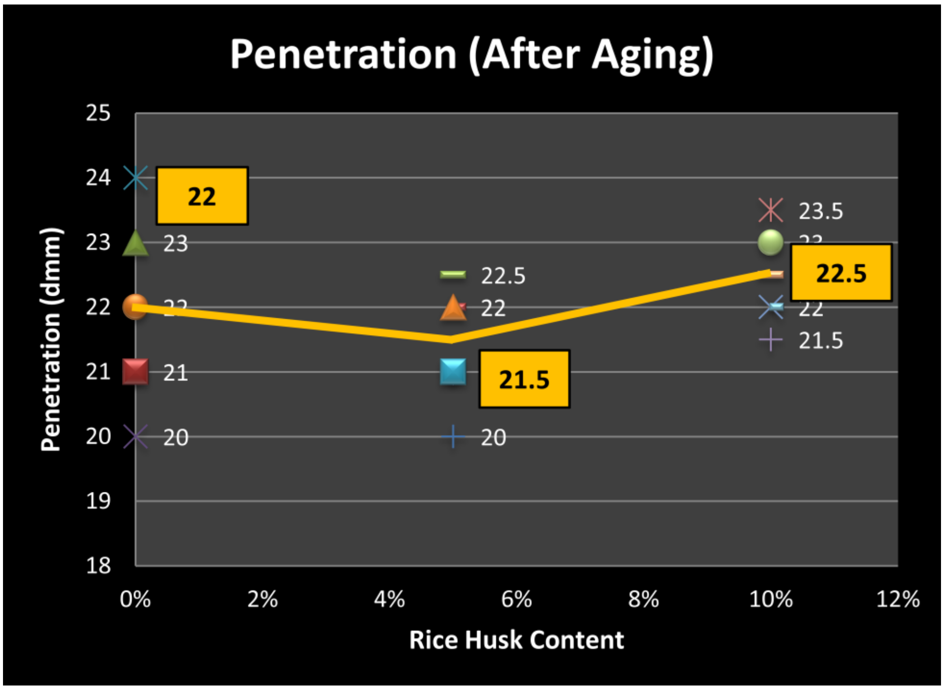

Figure 4.6: *Penetration (After Aging) versus addition of rice husk*

4.6 Softening Point Test (After Aging) Results

The softening point of every sample (virgin, 5%, and 10%) after the aging process have been successfully obtained. Note that only 2 sets of experiments (instead of three sets, due to time constraint) have been conducted for each of the sample in order to maximize the results' consistency as well as eliminating data that were out of range. The results were recorded as in **APPENDIX 4** and the graph showing the effects of adding different amount of rice husk powder is as shown in **Figure 4.7**.

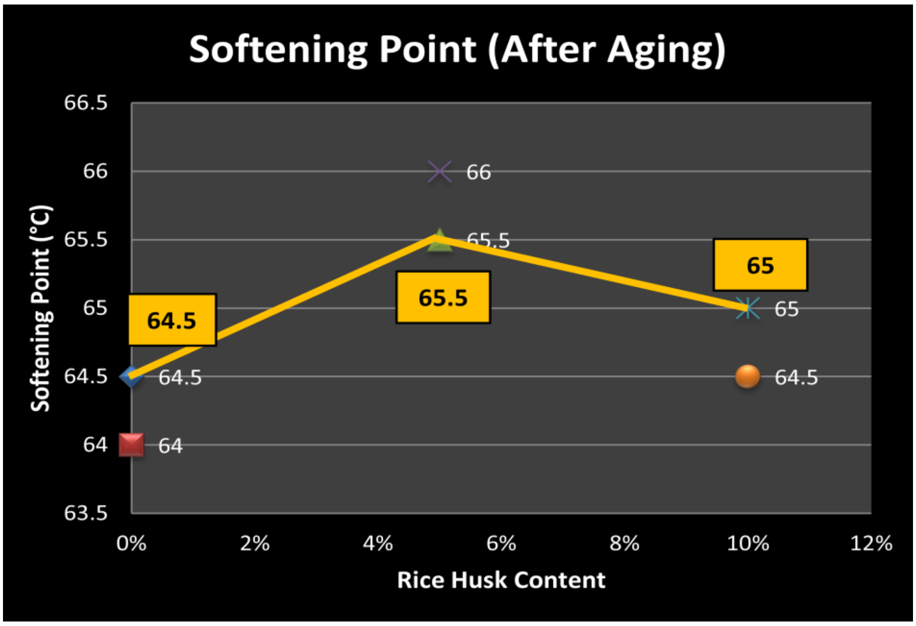

Figure 4.7: *Softening Point (After Aging) versus addition of rice husk*

4.7 Percentage Reduction of Penetration After Aging

The average penetration data (before and after aging) of all samples were analyzed and compared. The graph showing percentage reductions of penetration value after the aging process are as shown in **Table 4.3** and **Figure 4.8**.

Table 4.3: *Percentage Reduction of Penetration After Aging*

Rice Husk Content	Average Penetration (decimillimetre)		Percentage Reduction (%)
	Before Aging	After Aging	
0%	89.5	22	-306.8
5%	84.5	21.5	-293.0
10%	84	22.5	-273.3

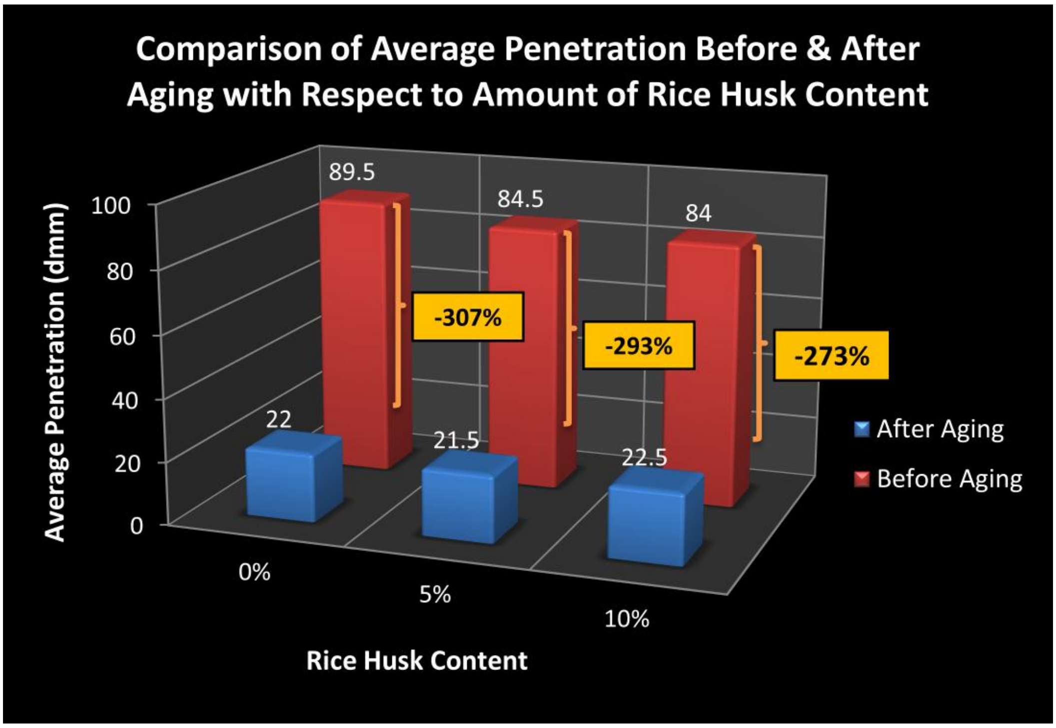

Figure 4.8: *Percentage Reduction of Penetration After Aging*

4.8 Percentage Increment of Softening Point After Aging

The average softening point data (before and after aging) of all samples were analyzed and compared. The graph showing percentage increments of softening point value after the aging process are as shown in **Table 4.4** and **Figure 4.9**.

Table 4.4: *Percentage Increment of Softening Point After Aging*

Rice Husk Content	Average Softening Point (°C)		Percentage Increment (%)
	Before Aging	After Aging	
0%	47	64.5	27.1
5%	49	65.5	25.2
10%	49	65	24.6

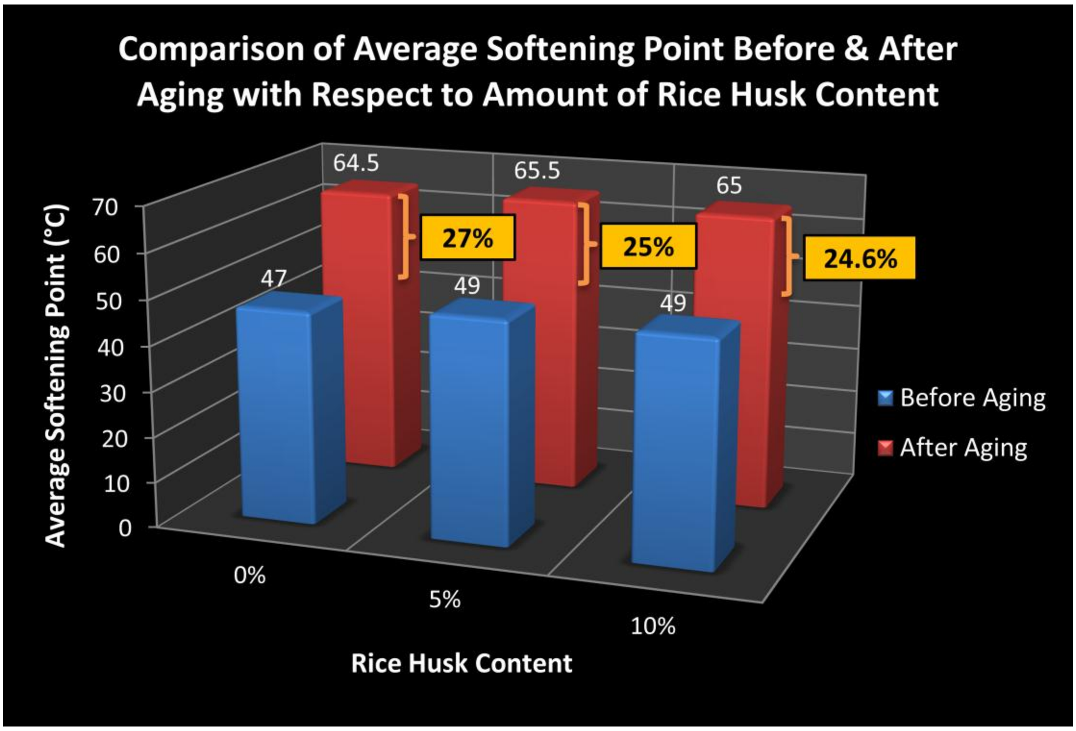

Figure 4.9: *Percentage Increment of Softening Point After Aging*

CHAPTER 5

DISCUSSION

From the results of Physical Test (before and after laboratory aging process) of bitumen samples that contain different amount of rice husk powder (0%, 5%, and 10%), there are significant trends that can be observed and analyzed.

5.1 Observations on Physical Properties Changes (Before Aging)

During the mixing process of rice husk powder with virgin bitumen, the evidence of physical properties changes (outer appearance) can straight away be noted. The texture of bitumen containing rice husk powder varies significantly as compared to normal virgin bitumen on which the one with rice husk possess more ‘rugged’ surface. Changes in the smell of the bitumen containing rice husk powder are also evident on which indicating a successful reaction between the bitumen and the rice husk powder.

Another physical differences between both virgin and rice husk bitumen is the ability to be stretched a distance before it breaks into two, on which the latter failed to be stretched a greater distance before it breaks. This indicates the reduction to some degree of the ductility and flexible properties of the bitumen after it has been mixed with rice husk powder.

It is also evident that, in comparison, for the same amount of virgin bitumen and rice husk bitumen, with the same heating temperature (100°C), the bitumen containing rice husk powder has to be heated in the oven relatively a longer period of time in order to achieve the pouring state (in almost liquid form). Accurate amount of time needed for heating have not been recorded, but it is estimated that the bitumen containing rice husk powder have to be heated for about an extra of 15 to 20 minutes in relative to normal virgin bitumen in order to achieve pouring state. Not only that, the time lag of cooling period from pouring state to solid state is also evident on bitumen that contains rice husk powder (about 20 to 25 minutes more). This early observation indicates that the bitumen containing rice husk powder is less susceptible to temperature changes as compared to normal virgin bitumen.

All and all, the physical property changes on virgin bitumen after mixing it with rice husk powder can only be accurately described based on the laboratory tests data, in this case, based on the Laboratory Physical Tests (Penetration Test and Softening Point Test) data.

5.2 Observations on Physical Properties Changes (After Aging)

There are some significant differences between virgin bitumen and bitumen that contain rice husk. This is as evident in **Figure 5.1** (Virgin Bitumen), **Figure 5.2** (5% Rice Husk Bitumen) and **Figure 5.3** (10% Rice Husk Bitumen). Note that the figures shown are the samples that were just aged by PAV. The bitumen now possessed a lifespan of similar of about 8 to 10 years of in-service life. The major physical change that differentiated the three samples from each other is the arrangement of air bubbles on its surface.

Figure 5.1: *Virgin bitumen's surface (After Aging)*

Figure 5.2: *5% Rice Husk bitumen's appearance (After Aging)*

Figure 5.3: *10% Rice Husk bitumen's appearance (After Aging)*

The virgin bitumen possessed the arrangement such that only in certain specific area that the bubbles accumulate, leaving a portion that does not have any air bubble. The diameters of air bubbles in certain part of the surface are also large (about 1 to 2.5 centimeters). This arrangement may due to the degree of hardness that the aged bitumen possessed that the air bubbles are inhibited to 'roam' freely throughout the bitumen.

Meanwhile, both bitumen samples that contain 5% and 10% rice husk content showed a uniformly distributed air bubbles arrangement throughout the bitumen surface. The diameter of all air bubbles is also very small (around 1 to 2 millimeters). This indicates that the hardness of bitumen containing rice husk might be lower than the virgin bitumen, thus allowing air bubbles to 'roam' freely throughout its surface.

Other physical changes can be evident from the amount of time needed for heating before the bitumen is fluid enough to pour. From the observation, the rice husk bitumen is relatively quicker to be heated until fluid compared to the virgin bitumen sample. It is also evident that the bitumen containing rice husk is slightly softer and easier to work with during Physical Test (After Aging), indicating that the bitumen is, in relative to virgin bitumen, still soft and flexible even after aging process.

5.3 Penetration Data Analysis

5.3.1 Penetration Before Aging

From the Penetration Tests (Before Aging) conducted on three sets of bitumen samples containing 0%, 5%, and 10% rice husk powder, the results (as shown in **Figure 4.1**) indicate that the 10% rice husk content bitumen scored the least penetration grade with an average value of 84 dmm, followed by 5% rice husk content bitumen, 84.5 dmm, and 89.5 dmm for pure virgin bitumen which is the largest value among all samples.

It is obvious that there are changes in physical properties of bitumen in term of penetration grade after being mixed with rice husk powder. The relatively lower penetration grade of bitumen that contain rice husk indicates that the bitumen became harder as compared to pure virgin bitumen.

In comparison, the penetration grade of 5% and 10% rice husk content bitumen is about the same (the value does not deviate much). This indicates that there is a certain limitation of amount of rice husk needed in order to achieve the best mixing content (ratio between bitumen and rice husk powder) for the bitumen to perform at its best performance during application.

5.3.2 Penetration After Aging

From the Penetration Tests (After Aging) conducted on three sets of bitumen samples containing 0%, 5%, and 10% rice husk powder, the results (as shown in **Figure 4.6**) indicate that the 5% rice husk content bitumen scored the least penetration grade with an average value of 21.5 dmm, followed by virgin bitumen at 22 dmm and the highest recorded penetration value is scored by the 5% rice husk content bitumen which is 22.5%.

It is determined that the 5% rice husk bitumen have the highest degree of hardness as compared to the other samples while the 10% rice husk bitumen have the softest degree of hardness in term of penetration value.

5.4 Softening Point Data Analysis

The data from softening point tests are crucial in a way that it certified the penetration data of bitumen. By right, the softening point data should be logical with respect to the penetration data, for example, if a bitumen sample possess low penetration grade, its softening point must be high, as both low penetration grade and high softening point indicates a hard bitumen (and vice versa to soft bitumen).

5.4.1 Softening Point Before Aging

From the tests results (as shown in **Figure 4.2**), the highest softening point temperature is scored by both 5% and 10% rice husk content bitumen and which is 49°C, followed by the least softening point temperature of 47°C scored by pure virgin bitumen.

It is obvious that there are changes in physical properties of bitumen in term of softening point after being mixed with rice husk powder. The relatively higher softening point value of bitumen that contain rice husk indicates that the bitumen became harder as compared to pure virgin bitumen.

5.3.2 Softening Point After Aging

As shown in Figure 4.7, the highest softening point value recorded after the aging process is 65.5°C which is scored by the 5% rice husk bitumen. This is followed by the 10% rice husk bitumen with recorded softening point of 65°C. The lowest softening point is 64.5°C which is the softening point of virgin bitumen.

It is determined that the 5% rice husk bitumen have the highest degree of hardness as compared to the other samples while the virgin bitumen have the softest degree of hardness in term of softening point value.

5.5 Temperature Susceptibility and Penetration Index Analysis

Both Temperature Susceptibility value and Penetration Index of bitumen derived from the penetration and softening point data (before aging) are crucial in order to assess the effects of rice husk powder on quality of virgin bitumen against exposure to temperature changes. For example, low temperature susceptible bitumen with high penetration index value indicates that the bitumen is more difficult to respond to temperature change such as from low temperature to high temperature, causing it to consume relatively longer time to become pouring state from solid state (as compared to higher temperature susceptible bitumen).

From the results (as shown in **Figure 4.4** and **Figure 4.5**), it is evident that the 5% rice husk content bitumen is the lowest temperature susceptible bitumen with the A-value of 0.0407 and the highest penetration index of -0.1115, indicating the highest degree of difficulty for it to respond to temperature changes. This is followed by 10% rice husk content bitumen with A-value of 0.0408 and penetration index of -0.1290, while the highest temperature susceptible bitumen sample is the pure virgin bitumen with A-value of 0.0432 and penetration index of -0.5123.

These data have proven that the rice husk powder can affects bitumen quality in term of low susceptibility to temperature changes. This is an utterly important characteristic that makes it more reliable for the application in road construction as compared to current virgin bitumen that is usually more susceptible to temperature changes. Theoretically, low temperature susceptible bitumen can last longer during in-service period as it does not react rapidly with high temperature from the heat of sun radiation and traffic tire frictions that are the major causes of aging problem in current road pavement. As the bitumen consume longer time to react with temperature changes, the oxidation reaction can also be slowed down in relative to normal current bitumen used for HMA road construction.

5.6 Volatiles Lost During RTFO Aging Process

It has been determined that all bitumen samples that were RTFO-aged were losing their weights after the aging process. As shown in **Figure 4.3**, the virgin bitumen lost the highest percentage of its volatiles which is 7.2%, followed by 5% rice husk bitumen with 6.4% weight reduction and 5.6% for 10% rice husk bitumen. This shows a significant improvement in terms of preventing mass volatiles lost in the samples that contain rice husk, on which, the 10% rice husk bitumen showed the best performance. The antioxidant material (rice husk) has managed to inhibit aging process and allows the bitumen to last longer (and not easily brittle) as compared to the virgin bitumen, thus another strong point to confirm the project’s objective.

5.7 Antioxidant’s Effectiveness (The Verdict)

From the graph shown in **Figure 4.8**, in comparison between the penetration grade before and after the aging process, all bitumen samples have degrade and have lost its soft properties (hardness increase). The percentage reduction of virgin bitumen is the highest which is -307%, from 89.5 dmm to 22 dmm. This is followed by the reduction of penetration for 5% rice husk bitumen, from 84.5 dmm to 21.5 dmm which is a -293% of reduction. The least percentage reduction of penetration is scored by the 10% rice husk bitumen, on which the penetration value reduces from 84 dmm to 22.5 dmm.

Meanwhile, the trends are also the same for the softening point percentage increment. It is recorded as in Figure 4.9 that the highest percentage increment of softening point is the virgin bitumen with 27% increment, from 47℃ to 64.5℃. This is followed by the 5% rice husk bitumen with 25% increment, from 49℃ to 65.5℃. The lowest percentage increment is 24.6% on which the softening point of 10% rice husk bitumen increases from 49℃ to 65℃.

The results showed the most effective bitumen in term of preventing aging and loss of hardness (on which makes it brittle) is the bitumen that contains **10% rice husk** content, followed by 5% rice husk content, and the worst is the virgin bitumen (0% rice husk). This is due to the highest recorded percentage reduction of penetration and the lowest percentage increment of softening point that indicates the prevention of bitumen from hardening effects as much as the other samples. It can be concluded that the 10% rice husk bitumen does not degrade significantly as compared to the other samples as it still maintain its flexibility and low hardness percentage reduction even though it has been aged completely (short-term aging and long-term aging).

Plus, the 10% rice husk bitumen also scored the lowest percentage of mass quantity of volatiles lost during RTFO aging. Since RTFO simulates short-term aging; the condition of road construction period during which the bitumen was exposed to high temperature and atmospheric oxygen; the 10% rice husk bitumen is deemed to able to maintain its molecular interlocking states and able to prevent further volatiles lost better than virgin bitumen.

All of the obtained results throughout this project have managed to fortify the hypothesis that the rice husk did possess an antioxidizing property that inhibits the reaction between free radicals compounds (by-product of aging) with asphaltene in bitumen, thus preventing aging, and consequently gives an extended lifespan to the bitumen as compared to the virgin bitumen that does not contain rice husk.

5.8 Problems/Constraints Faced & Remedial Actions Taken

For this project, the major issue was the delay for conducting the Laboratory Aging Test as the aging machines namely Rolling Thin-Film Oven (RTFO) and Pressure Aging Vessel (PAV) are not fully safe to be used due to wiring/electrical problems. It has been notified by the Lab Technicians that the machine would take a minimum of three weeks to be repaired before it is fully safe to be operated for conducting the aging test.

In order to achieve the most reliable data for this project as stated in **Chapter 4: Results & Calculations**, there has been numerous times of repetition/redo of laboratory test on penetration and softening point determination as unreliable results that are usually out of range and illogical have been obtained due to lack of laboratory skills.

There are also conflicts in term of time-constraint, as more samples have not been able to be tested in order to achieve more accurate results, especially during Physical Test After Aging, on which only two penetration cups and 2 ring-and-balls apparatus were prepared for every bitumen samples (0%, 5%, and 10%) for the penetration and softening point determinations. The limited time-constraint has also affected the conduct of any required repetition/redo of the Physical Test after aging. This could greatly affect the results and would be a total reliability if the current obtained results are not as accurate as it should be.

CHAPTER 6

CONCLUSION & RECOMMENDATION

6.1 Rice Husk Potential as Bitumen Modifier

The results obtained from the laboratory tests conducted and the analytical analysis of all data have managed to fortify the hypothesis that rice husk do possess the potential to reduce volatiles lost during short-term aging (which is during road construction period), and contributes to reduce the rate of bitumen degradation (hardening effect) due to aging process, thus extending its lifespan.

It has been observed and analyzed that the antioxidant material (rice husk powder) also has the potential to improve current virgin bitumen quality in term of low susceptibility to temperature changes.

It can be concluded that the best sample in term of reducing aging effect is the 10% RHB which scored the least percentage reduction of penetration and the highest percentage increment after being short-term aged (RTFO) and long-term aged (PAV). Moreover, the 10% RHB also scored the least amount of volatiles lost which is 5.6% of weight reduction after the short-term aging (RTFO) process. Even though it does not possess the lowest temperature susceptibility (as compared to 5% RHB), the value is still promising as it still is lower than the virgin bitumen (0% RHB). Low temperature susceptibility is one of the main characteristics that slow down aging, as the bitumen consume more time in order to complete oxidation reaction. Thus, the bitumen lifespan increases, and by this means, maintenance cost can be saved.

Overall, the data indicates that the sample containing rice husk have the ability to slow down aging by scavenging free radicals exist in bitumen. Asphaltene compound has not been deteriorated to a much degree compared to the virgin bitumen (0% RHB). Not only that, Rice Husk Modified Bitumen is also easier to produce as compared to Polymer Modified Bitumen, thus cheaper investment on production cost. The utilization of rice husk (waste material) for industrial benefits is also an act towards promoting sustainable development.

6.2 Recommendation

For even achieving better results for this project, the number of bitumen samples containing different amount (percentage) of rice husk powder can also be increased. For example, an extra of two samples containing 3% or 7% rice husk powder content should be prepared in order to determine the most optimum rice husk content that should be mixed with virgin bitumen in order to perform at its best performance during in-service period of HMA-paved roadways application.

In the pursuit of obtaining the most accurate results for this project, it is a must to not solely rely on physical tests' results to conclude the degree of effectiveness of antioxidant in bitumen. **Chemical Tests** have to be conducted on the bitumen samples in order to achieve a higher degree of accuracy results and have better in-depth understandings of the rheological properties of bitumen that reacts with lignin compound found in rice husk. For that matter, Fourier Transform Infrared spectroscopy (FTIR) test and Dynamic Shear Rheometer (DSR) test have to be taken into account for the sake of perfecting this project.

In the later future (beyond FYP context), elaborate research regarding the suitability of antioxidant powder, not only as bitumen modifier, but also as filler material in hot mix asphalt production have to also be tested. Plus, instead of only researching on rice husk material, the suitability of other materials such as coconut shell and palm shell as antioxidant material should also be determined. The research data on that matter may be very useful for asphalt construction, as not only the said antioxidant materials can prolong the service life of asphalt pavement, but also, the application of environmental friendly filler material (as compared to Portland Cement) is an added advantage towards the current asphalt production industry.

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APPENDICES

APPENDIX 1: PENETRATION (BEFORE AGING) RESULTS

APPENDIX 2: SOFTENING POINT (BEFORE AGING) RESULTS

APPENDIX 3: PENETRATION (AFTER AGING) RESULTS

APPENDIX 4: SOFTENING POINT (AFTER AGING) RESULTS

APPENDIX 1

PENETRATION (BEFORE AGING) RESULTS

STANDARD PENETRATION TEST (BEFORE AGING)						
BS2000: Part 49: 1983 / ASTM D5						
Temperature: 25°C, Load: 100 gram, Time: 5 seconds						
Virgin Bitumen Sample (0% RHB)						
Cone No.	Determination 1	Determination 2	Determination 3	Mean (dmm)	Difference	Comments
1	87	88.5	90	88.5	3	Okay
2	90.5	91.5	94	92.0	3.5	Okay
3	88.5	89.5	93	90.3	4.5	Reject
5% Rice Husk Bitumen Sample (5% RHB)						
Cone No.	Determination 1	Determination 2	Determination 3	Mean (dmm)	Difference	Comments
1	83	84.5	82	83.2	2.5	Okay
2	83	85	83.5	83.8	2	Okay
3	82	83.5	86	83.8	4	Okay
10% Rice Husk Bitumen Sample (10% RHB)						
Cone No.	Determination 1	Determination 2	Determination 3	Mean (dmm)	Difference	Comments
1	83.5	84.5	90	86.0	6.5	Reject
2	83	85	84	84.0	2	Okay
3	83.5	85.5	86	85.0	2.5	Okay

APPENDIX 2

SOFTENING POINT (BEFORE AGING) RESULTS

SOFTENING POINT TEST (BEFORE AGING)						
BS2000: Part 58: 1983 / ASTM D36						
Virgin Bitumen Sample (0% RHB)						
Sample No.	Ball 1 (°C)	Ball 2 (°C)	Mean (°C)	Round to the nearest 0.2°C	Difference (°C)	Comments
1	46	47.5	46.75	46.6	1.5	Reject
2	48	48.5	48.25	48.2	0.5	Okay
3	46	46.5	46.25	46.2	0.5	Okay
5% Rice Husk Bitumen Sample (5% RHB)						
Sample No.	Ball 1 (°C)	Ball 2 (°C)	Mean (°C)	Round to the nearest 0.2°C	Difference (°C)	Comments
1	48.5	49.5	49	49	1	Okay
2	48	49	48.5	48.6	1	Okay
3	49	50	49.5	49.6	1	Okay
10% Rice Husk Bitumen Sample (10% RHB)						
Sample No.	Ball 1 (°C)	Ball 2 (°C)	Mean (°C)	Round to the nearest 0.2°C	Difference (°C)	Comments
1	49	50	49.5	49.6	1	Okay
2	47.5	48	47.75	47.8	0.5	Okay
3	48.5	49	48.75	48.8	0.5	Okay

APPENDIX 3

PENETRATION (AFTER AGING) RESULTS

STANDARD PENETRATION TEST (AFTER AGING)						
BS2000: Part 49: 1983 / ASTM D5						
Temperature: 25°C, Load: 100 gram, Time: 5 seconds						
Virgin Bitumen Sample (0% RHB)						
Cone No.	Determination 1	Determination 2	Determination 3	Mean (dmm)	Difference	Comments
1	21	21	23	21.7	2	Okay
2	20	24	22	22.0	4	Okay
5% Rice Husk Bitumen Sample (5% RHB)						
Cone No.	Determination 1	Determination 2	Determination 3	Mean (dmm)	Difference	Comments
1	20	19	19.5	19.5	1	Okay
2	18	19	18.5	18.5	1	Okay
10% Rice Husk Bitumen Sample (10% RHB)						
Cone No.	Determination 1	Determination 2	Determination 3	Mean (dmm)	Difference	Comments
1	19.5	18	20	19.2	2	Okay
2	17.5	19	18	18.2	1.5	Okay

APPENDIX 4

SOFTENING POINT (AFTER AGING) RESULTS

SOFTENING POINT TEST (AFTER AGING)						
BS2000: Part 58: 1983 / ASTM D36						
Virgin Bitumen Sample (0% RHB)						
Sample No.	Ball 1 (°C)	Ball 2 (°C)	Mean (°C)	Round to the nearest 0.2°C	Difference (°C)	Comments
1	64	65	64.5	64.6	1	Okay
2	64	64.5	64.25	64.2	0.5	Okay
5% Rice Husk Bitumen Sample (5% RHB)						
Sample No.	Ball 1 (°C)	Ball 2 (°C)	Mean (°C)	Round to the nearest 0.2°C	Difference (°C)	Comments
1	65	65.5	65.25	65.2	0.5	Okay
2	64.5	65	64.75	64.8	0.5	Okay
10% Rice Husk Bitumen Sample (10% RHB)						
Sample No.	Ball 1 (°C)	Ball 2 (°C)	Mean (°C)	Round to the nearest 0.2°C	Difference (°C)	Comments
1	65	66	65.5	65.6	1	Okay
2	65.5	66	65.75	65.8	0.5	Okay