# The Effect of Black Rice Husk Ash on the Rheological Properties of Bitumen

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**Abstract.** Black rice husk ash (BRHA) waste product is cheaper and can be obtained from the rice mill. Reused waste product can ideally reduce pollution problem due to disposal aspect. The commercial value of BRHA was increase and suitable used in road construction. In this study, BRHA waste was grind using grinding ball mill for 120 minute to form fine powder. Then BRHA is sieve to size less than 75 µm. At laboratory, BRHA was mixed with bitumen to replace 2%, 4%, 6 % and 0% represent as control sample. The penetration, softening point, DSR, and RTFO was performance in this investigation. The results show that the bitumen becomes more harder, while rate of penetration decrease when replacement BRHA amount increase. Softening point test of bitumen also increased. The result also indicates that modification of bitumen can relieve the effect of aging due to short term aging test. BRHA waste improves the performance of bitumen when it was added into bitumen. It can be concluded that usage of BRHA helps to improve the performance of the road pavement which also reduces the rutting effect.

## Introduction

Road structure have deteriorated more rapidly in recent years because of the increase in traffic volume, loading and poor maintenance. The common causes ofpavement deterioration and degradation are overloading, seepage, improper or poor road surface drainage, lack of proper road maintenance, lack of proper design, adverse climatic conditions and some other factors. Identification of the road cracks at an early stage is essential as preventive road maintenance and effective remedial measures can be applied before the problem becomes too severe and the pavement fails. To minimize such deterioration and increase the long term durability of a flexible pavement, bituminious layers must be improved. All the rage to array an alternative which could reduce the deterioration of the roads, the use of industrial waste materials has been widely studied for seeking suitability utilized in road construction.

Black rice husk ash (BRHA) is one of the agro-waste that could be used on the rheological properties of bitumen. Rice husk can be found as natural materials, this material is also obtained with requiring low cost, energy and time. Unfortunately BRHA waste material is dumped into environment without any commercial return.

The burning of rice husk under controlled temperature produces a highly reactive black rice husk ash (BRHA). In general, the quality from BRHA depend on: (a) the content Silica achieved by complete combustion of rice husk, (b) an amorphous silica phase crystallization formed by the combustion of suitable rice husk on temperature control or un-control and treatment of BRHA, and (c) the size and surface area of the ash particles obtained by the grinding process.

The concept of sustainable development, currently became a very hot issues, requires that the society as a whole become aware of the necessity to make the most of all existing resources, trying to minimize creation of residues [2]. Is necessary to address the engineering, environmental and economic concerns before using the industrial by product BRHA in orders to achieve sustainable development.

## Previous Studies

The burning of rice husk under uncontrolled temperature produces a highly reactive black rice husk ash (BRHA). Utilization of industrial waste such as BRHA as modifier in bitumen could solve waste problem. Black rice husk ash produced activated carbon. Material with activated carbon usually found from coconut shell, palm oil fuel ash, coal and also rice husk ash (RHA). Material with activate carbon having highly developed internal surface area and high porosity. Some review of research result to understanding the rheological properties of Rice husk ash.

Effect RHA as cement and fine aggregate have a good result, RHA was burnt for collected the porous as fine aggregate to mixed in cement, The workability of RHA concrete have decreased if the percentage of replacement increases[3]. Modification of bitumen is possible during different stages of its usage, eitherimmediately before paving mix production or in between binder production and mixproduction[4].BRHA was used to replace OPC (Ordinary Portland cement), the result showed that the early age (during the first 7 days of curing) of strength development in cement blended with BRHA was lower compared with the controlled cement paste, but thereafter, the high strength was development during 14 to 91 days of curing[5].

Strength of mortar and concrete as influenced by RHA also review by researcher [6]. Advantages and disadvantages of supplementary use of RHA in mortar or concrete are mentioned in the journal, the critical review on the influences of RHA on the strength of mortar and concrete are mainly presented. The strength that development of concrete produced with a particular level of RHA replacement is the same or higher as compared to OPC concrete.

## Methodology

Study was be carried out by using experimental methods to evaluate the effect of BRHA in bitumen. The sample testing on BRHA that was conduct is show in Figure 1.

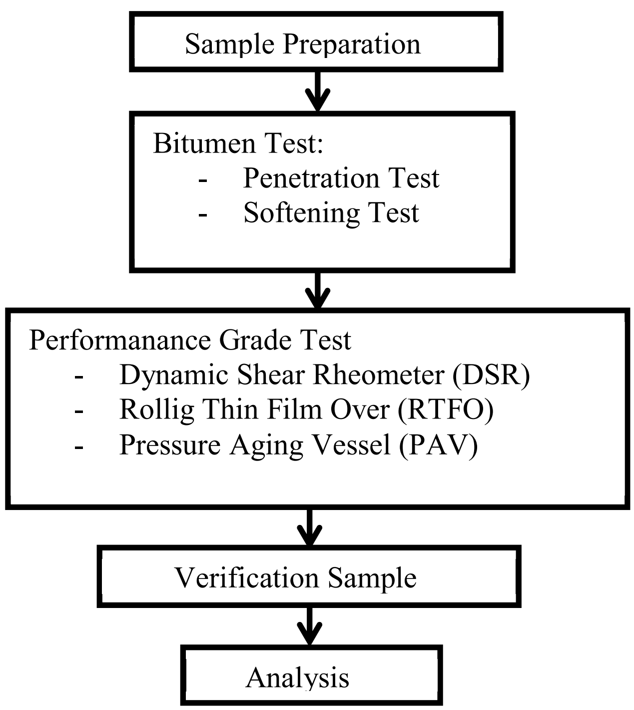


Figure 1: Flow chart of methodology

## Material and Testing Method

***Materials Used***Bitumen with 60/70 PEN grade was used in this study as recommended in new JKR Standard Specification for Road Works (2007) based on tropical climate. The asphalt exhibits the following physical properties: penetration 69 d-mm at 25 ˚C (ASTMD5-97), penetration after short term aging 49 d-mm at 25 ˚C; softening point, 52 ˚C (ASTMD36-95); DSR G*/sinδat 70 ˚C is 0.786 (kPa) and after RTFO ASTM D 2872 (Short term aging) DSR at 70˚C is 1.232 (kPa).

***Materials Preparation*** For sample preparation, waste material modified with asphalt for this study is Black Rice husk ash (BRHA). Black rice husk ash in this experiment was bought from factory in Muar. The burning of rice husk under uncontrolled temperature produces a highly reactive black rice husk ash (BRHA) that produce activated carbon. In this experiment BRHA is fully dried.BRHA waste will be grind using grinding ball mill for 120 minute to form fine powder. Then BRHA is sieve to form size (less than 75 µm). BRHA was mixed with bitumen to replace 2%, 4% and 6% they are name as BRHA-2, BRHA-4, BRHA-6 and BRHA-0 represent control sample (unmodified bitumen).

***Binder Testing***The bitumen with 60/70 PEN grade was first heated until it became well-melting fluid at approximately 160 ˚C (temperature range from 135 ˚C to 165 ˚C). After the bitumen melt, BRHA is added to replace bitumen at 2%, 4% and 6 % by total weight of bitumen Mixing of bitumen and BRHA is carried out using high shear mixer (Figure 2). Certainly, the mixture is blended at 800 rpm for 60 minutes to ensure uniform dispersion of BRHA in bitumen.

The number of sample require are:

1. 4 samples set of samples to be prepared in this experiment. Where each set will modified bitumen with replacement of BRHA for each percentage 0%, 2%, 4% and 6 %. (Penetration Test) ASTMD5-97;
2. 4 samples set of samples to be prepared. Where each set will modified bitumen with replacement of BRHA for each percentage 0%, 2%, 4% and 6 %. (Softening Point Test)ASTMD36-95;
3. 4 samples set of samples to be prepared in this experiment. Where each set will modified bitumen with replacement of BRHA for each percentage 0%, 2%, 4% and 6 %. (DSR Test);
4. 8 samples set of samples to be prepared for Aging Test. Where each 2 set will modified bitumen with replacement of BRHA for each percentage 0%, 2%, 4% and 6 %. (RTFO Test)ASTM D 2872;
5. 4 samples set of samples from RTFO Test to be prepared for Aging test. That will be compared with Un-aged test (Penetration Test)ASTMD5-97;
6. 4 samples set of samples from RTFO Test to be prepared for Aging test. That will be compared with Un-aged test (DSR Test);
7. 


Figure 2: high shear mixer

***Penetration Test*** Penetration Test(ASTMD5-97) is simple and easy to perform but it does not measure any fundamental parameter and with one temperature 25˚C (77˚F). This test is used for evaluating the consistency of bitumen before and after heating. The objective of this test is to measure the penetration value of bitumen.The apparatus used in this test are penetration apparatus, penetration needle, penetration cup, water bath, transfer dish, thermometer, timing device, heater and also balance.

***Softening Point Test*** Softening point (ASTMD36-95) is the temperature at which a substance attains a particular degree of softness under specified conditions of test. As temperature increases, asphalt cement changes from solid to liquid, and the stiffness of asphalt cement will reduce accordingly. Before mixing with aggregates to form a road pavement, asphalt cement must be soft enough in order for it to be handled easily during pavement work. The most common method to soften the asphalt cement is by heating it. Higher-grade asphalt cement has higher softening temperature compare to lower grade asphalt cement. The ring and ball test is commonly used to determine the softening temperature of asphalt cement. Softening is also considered as a measure of softness of the bituminous material.

***(DSR) Dynamic Shear Rheometer Test*** This test used to apply shear strain under a controlled temperature and frequency. And also use to characterize the viscous and elastic behavior of asphalt at high and intermediate services temperatures which can evaluate rutting and fatigue cracking potential of pavement. The test is conducted at a rotational frequency of 10 rad/sec (1.59Hz) and at the temperatures that correspond to the highest and intermediate temperature. The output of the test is the Dynamic Shear Modulus G* and the phase angle of the binder. The requirements are:

a. Original Binder: $G^*/\sin\delta > 1.0$ kPa to prevent rutting.
b. RTFO-aged binder: $G^*/\sin\delta > 2.2$ kPa to prevent rutting immediately after construction.
c. RTFO+PAV-aged binder: $G^*\sin\delta < 5000$ KPa to prevent fatigue cracking throughout the pavement life.

***(RTFO) Rolling Thin Film Oven*** Thin bottle with a small amount of asphalt placed in rack in an oven. To evaluate the effect of short term aged bitumen. Bitumen is exposed to manufacturing and placement in the meanwhile construction. RTFO also provides a quantitative measure of the volatiles loss when the aging process. Sample 35gr are poured into glass jars that rotate and turn in a heated oven at 163˚C/325˚F for 85 minutes. Figure 3 shows apparatus for RTFO test and glass jars .

Figure 3: RTFO ASTM D 2872 & Glass jars

**Data Analysis**

***Penetration*** Figure 4 shows the effect of Black Rice Husk Ash (BRHA) on the penetration value. The results indicated that for the un-aged bitumen, BRHA mix with 2%, 4% and 6 % replacement bitumen obtained penetration values of 67.8, 65.3 and 59.6 PEN, respectively. Meanwhile, 0% BRHA mix with 0 % replacement obtained 69 PEN. The addition of BRHA into bitumen directly reduces bitumen penetration. These results indicate that the penetration of the bitumen binder dramatically decreases with increasing BRHA content.

Meanwhile, all modified specimens subjected to short term aging showed a decrease in penetration. The control or 0% BRHA mix exhibited the highest penetration, whereas the 6% BRHA mix exhibited the lowest penetration. For examples, the penetration values were 65.3 and 59.6 PEN at 4% and 6% BRHA. Therefore, the penetration aging value decreases with increasing BRHA content. The results after short-term aging (RTFO) indicated that for the aged bitumen, BRHA mix with 2 %, 4% and 6 % replacement bitumen obtained penetration values of 46.8, 44.6 and 41.4 PEN. It is show in figure 5 for result after short-term aged (RTFO). These results suggest that all samples incorporated with BRHA. According to physical properties of Black Rice Husk Ash, it is expected that the Black Rice Husk Ash will harden the bitumen. The Modified Bitumen with Black Rice Husk Ash become more viscous and harder. Thus, BRHA can improve the performance properties of bitumen.

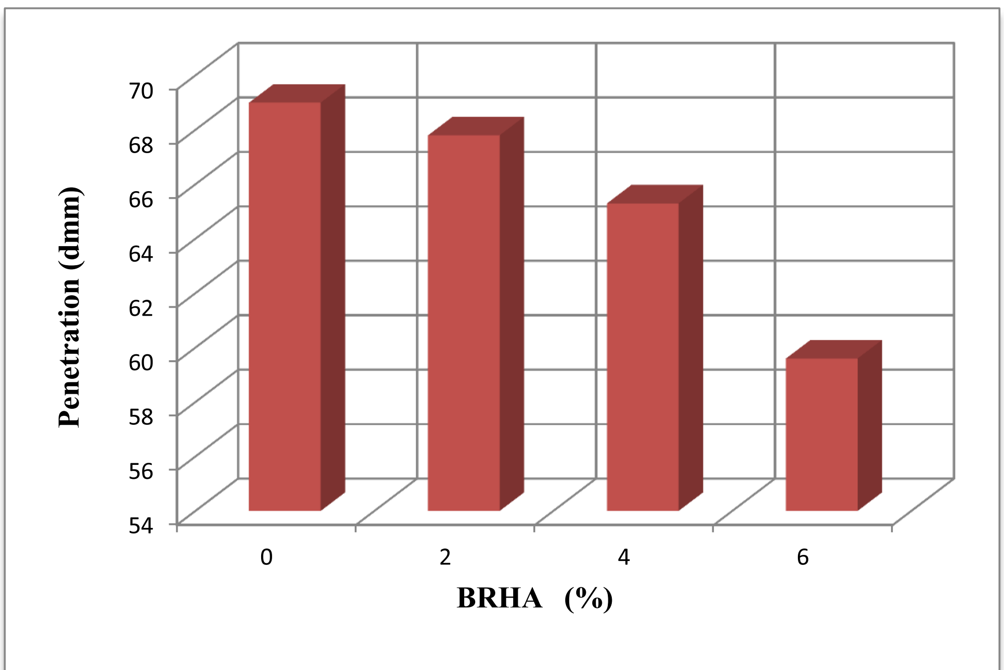


Figure 4: Penetration Result for bitumen 60/70 added BRHA (Un-aged)

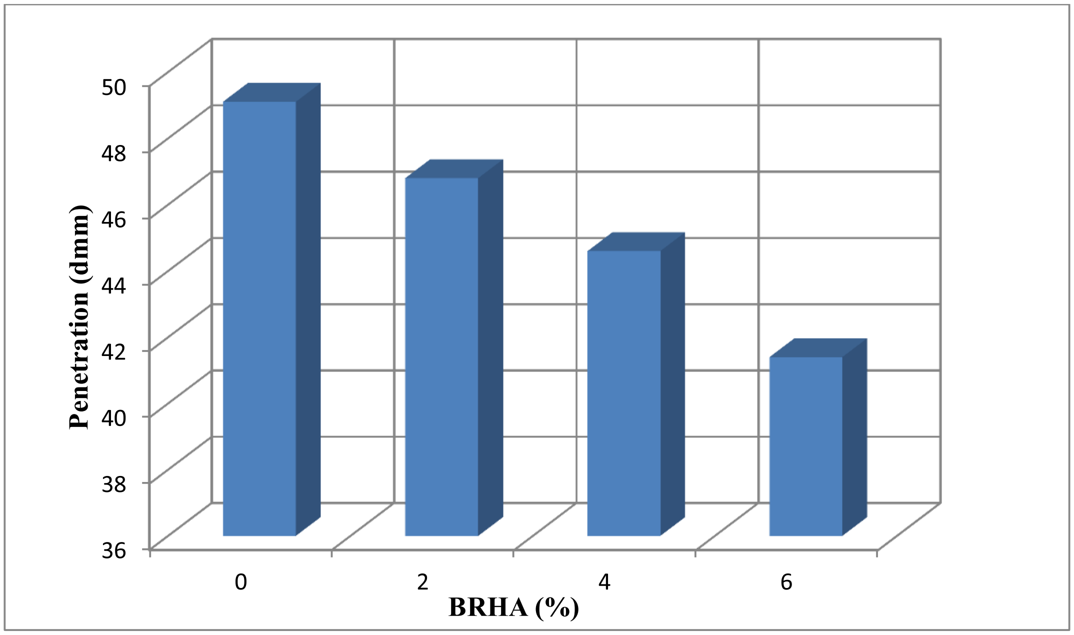


Figure 5: Penetration Result for bitumen 60/70 added BRHA (Aged)

***Retained Penetration***One option to show the effect of aging on the properties of bitumen is through penetration aging ratio. RTFO test was conducted to evaluate effect on rheological properties of unmodified and modified bitumen after short term aging. Penetration measures the bitumen consistency. Penetration aging ratio can be determined using equation 1:

$$\% \text{ Retained Penetration} = \frac{Penetration_{\,of\ RTFO}}{Penetration_{\,of\ unaged}} \times 100 \qquad \text{Equation 1}$$

After RTFO test, penetration of bitumen changes significantly, it show in Figure 5. Aging influences the percent retained penetration values of the BRHA-bitumen, as presented in Table 1. As shown in the table, the incorporation of BRHA in higher quantities decreased the retained penetration. For instance, the retained penetration was 69.03% at 2% BRHA content. However, the retained penetratin decreased to 68 % when the BRHA content increased to 6%. The tendency to age harden decreases with increases retained penetration. This result can be attributed to the obstruction of BRHA to the hardening process of the bitumen. The results also show that the addition of BRHA to the bitumen can reduce aging tendencies, as determined by the penetration test.

Table 1: Results of retained penetration (%) under RTFO

| **BRHA replacement (%)** | **Aging Test** | | |
|---|---|---|---|
| | **Pen Before RTFO** | **Pen After RTFO** | **Retained Pen (%)** |
| 0 | 69.0 | 49.1 | 71.16 |
| 2 | 67.8 | 46.8 | 69.03 |
| 4 | 65.3 | 44.6 | 68.30 |
| 6 | 59.6 | 42.4 | 68.0 |

***Softening Point Test*** Table 2 shows the softening point values for original and modified bitumen 60-70.

Table 2: Softening point values for original and modified bitumen 60-70

| % BRHA | Reading (˚C) | | Softening Point (˚C) |
|---|---|---|---|
| | Ball A | Ball B | |
| 0 | 51.8 | 52 | 52 |
| 2 | 53 | 52 | 52.5 |
| 4 | 52 | 54 | 53 |
| 6 | 54.6 | 54 | 54.3 |

Softening Point Test were done for normal bitumen (control sample) and modified bitumen with 2%, 4% and 6% of Black Rice Husk Ash waste content. The result was shown in Figure 6. The relationship between the Black Rice Husk Ash waste added and the softening point of the bitumen are slightly increase as shown in Figure 6, Softening point slightly increased with the increased amount of BRHA added. For instance, at 2 % BRHA, the softening point value increased from 52.0 ˚C control sample ( 0 % BRHA ) to 52.5 ˚C. At 4 and 6 % BRHA the softening point value were 53.0 ˚C  and 54.3 ˚C. The increase of softening point value indicates the resistance of BRHA modified bitumen to the effect of heat. in DSR test will shows further the effect of heat in modified bitumen with BRHA

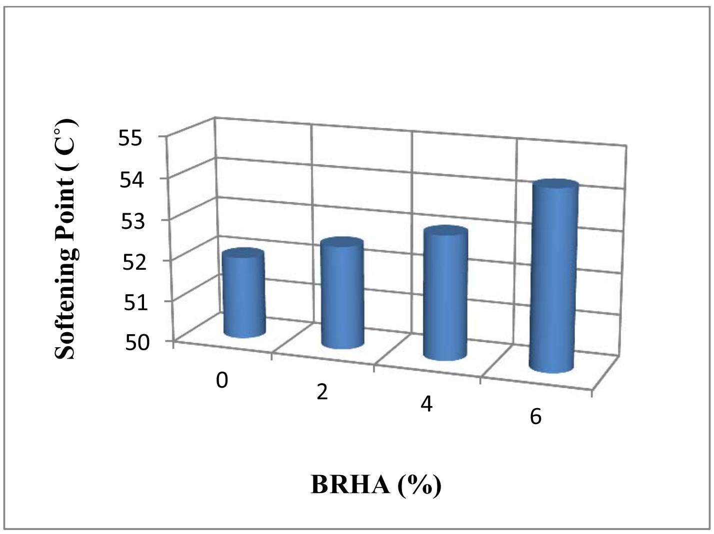


Figure 6: Softening point result for bitumen 60/70 added BRHA

***Penetration Index*** Penetration Index (PI) value is defined as the measure of temperature susceptibility of bitumen. Softening point (SP) together with Penetration (P) value is used to classify bitumen. PI shows the suitability of bitumen as pavement binder. The suitable penetration index for pavement construction is between -1 and +1. Table 3 shows the summary of the PI result. It can be determined using nomograph or the following equation 2:

$$Penetration\ Index\ (PI) = \frac{1952-500logP-20SP}{50\text{logP}-\text{SP}-120} \quad \text{Equation 2}$$

PI can also identify a particular type of bituminous material to a limited extent [6]. Table 3 lists the PI values for mixtures prepared at varying BRHA contents and exposed to different degrees of aging. As indicated in the table, the incorporation of BRHA content into the bitumen reduced the temperature susceptibility of the binder. Lower PI values indicate higher temperature susceptibility. Higher PI value indicate higher resistance to low-temperature cracking and permanent deformation [7].

Table 3: Penetration Index

| % RBHA | PI |
|---|---|
| 0 | 0.09 |
| 2 | 0.17 |
| 4 | 0.19 |
| 6 | 0.25 |

Table 3 shows the summary of PI Value for BRHA modified bitumen with different percentage of replacement. From the result that shows in Table 3, Penetration Index increased, From 0.09 to 0.25 when the amount of BRHA content 6 % in bitumen. The valued indicate it is in the range -1 to +1, which is suitable for pavement construction [8]. The penetration index slightly increased with the increased amount of BRHA added. The higher penetration index indicates higher resistance to low temperature cracking and permanent deformation.

***Direct Shear Rheometer Results*** This test used to apply shear strain under a controlled temperature and frequency. And also use to characterize the viscous and elastic behavior of asphalt at high and intermediate services temperatures which can evaluate rutting and fatigue cracking potential of pavement. The test is conducted at a rotational frequency of 10 rad/sec (1.59Hz) and at the temperatures that correspond to the highest and intermediate temperature. The output of the test is the Dynamic Shear Modulus G* and the phase angle of the binder. According to this study, at a given temperature, by increasing modified bitumen with 2%, 4% and 6% of Black Rice Husk Ash waste content, the G* value have increased. A similar style can be seen for modified bitumen after the RTFO aging process. In the specification, rutting is taken into account using a rutting factor (G*/ sin δ), which is solely dependent on the rheological properties of the asphalt binder. The higher the rut factor for the binder, the stiffer the asphalt concrete should be and thus more resistant to rutting [9].

Figure 7 and 8 shows the G*/ sin δ trend for original and modified bitumen, before and after short term aging process in a temperature ranging from 46 ˚C to 70 ˚C. From the Figure 7 it can be seen that in percentage 6 % BRHA the G*/ sin δ decrease above 4 and 2 % BRHA. Due to this result, the optimum BRHA that have good performance is 2% BRHA. The increase of G*/ sin δ means an improvement on the performance of modified bitumen against permanen deformation (rutting) at high temperature.

The loss stiffness (G*/ sin δ > 2.2 kPa) value of the aged binder is strongly associated with the fatigue life of the mixture and is a basic parameter for describing the fatigue characteristic of an asphalt binder. The increase in loss stiffness is accompanied by a rather significant decrease in fatigue resistance. Figure 8 show the result G*/ sin δ > 2.2 kPa in percentage 6 and 4 % BRHA were decrease above 2 % BRHA. From 2 graph Figure for DRS it shown that 2 % BRHA heve good performance againts rutting.

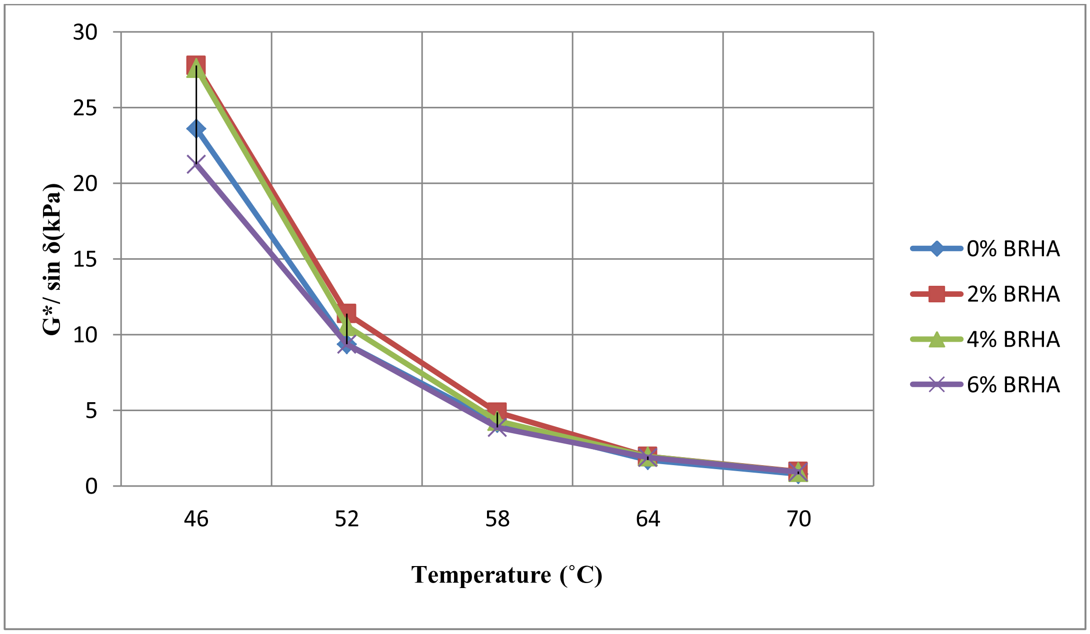


Figure 7: DSR Result (G*/ sin δ) of Bitumen 60-70 replacement with BRHA in Original State

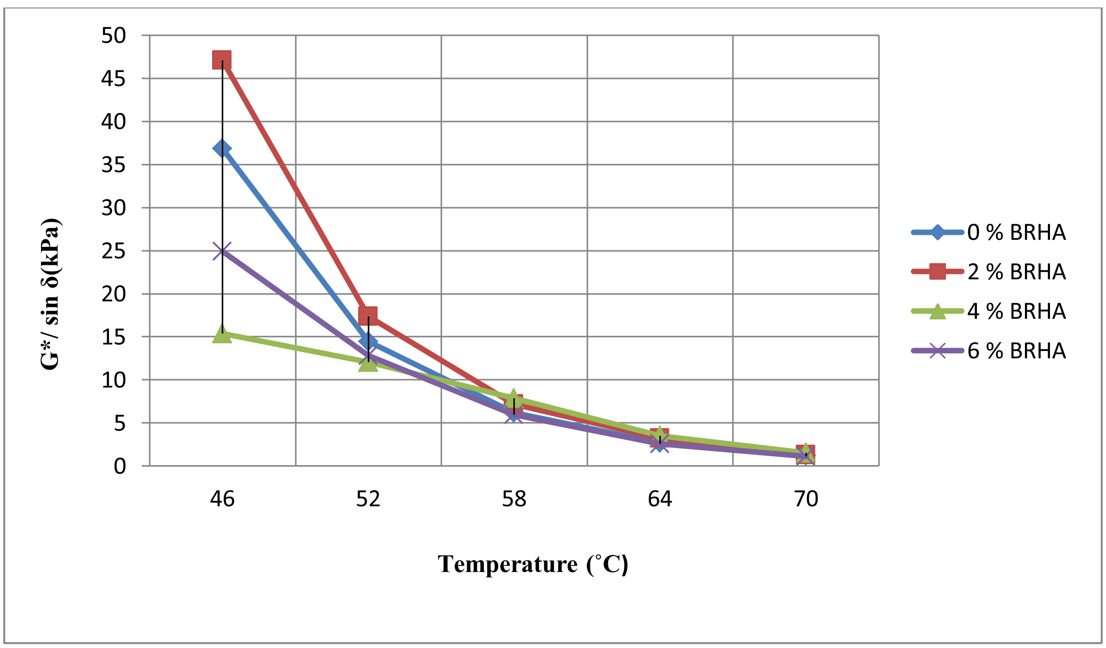


**Figure 8 –** DSR Result(G*/ sin δ) of Bitumen 60-70 replacement with BRHA in Short Term Aging

**Conclusion**

Black rice husk ash (BRHA) is one of the agro-waste materials that could be modifier with bitumen in highway construction. Bitumen with 60/70 PEN grade was used in this study as recommended in new JKR Standard Specification for Road Works (2007) based on tropical climate. The main objectives of this research are to investigate the effect of BRHA on the rheological properties of bitumen and to determine the optimum percentage of BRHA in bitumen. This can be completed by comparing the result of unmodified (control sample) and modified bitumen. Samples were prepared and tested, whether they meet the specification of JKR/SPJ/2007. Summaries of finding based on the analysis are:

1. Penetration Value of modifier bitumen with replacement of BRHA (2%, 4% and 6%) was decreased than normal bitumen. Furthermore after short term aging (RTFO) the result for penetration value was decreased compare to un-aged modifier bitumen.

2. Softening point slightly increased with the increased amount of BRHA added in bitumen, while the bitumen become more viscous as percentage of replacement was 6%.

3. Penetration index value for BRHA modified bitumen with different percentage of replacement slightly increased with the increased amount of BRHA added. The higher penetration index 0.25 indicates higher resistance to low temperature cracking and permanent deformation. The suitable penetration index for pavement construction is between -1 and +1.

4. BRHA has a great effect on the bitumen properties when replace into the bitumen before and after RTFO aging process. The increase in $G^*/\sin\delta$ means an improvement on the performance of modified bitumen against rutting at high temperature, but BRHA has diverse effect on cracking when the percentage of BRHA repalce in bitumen is greater than 2%.

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