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Cement and animal bone powder on rheological characteristics of bitumen

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Abstract. The developed countries have adopted the use of modifiers for assessment of asphalt binder long term performance. Asphalt contributes 90 to 95 % in cost estimation of Flexible pavement while compared to all other major material components. Since Ethiopia is one of the landlocked country without any fossils fuel source for the production of Asphalt, moreover Ethiopian importer depends on neighbour country Djibouti. This research analyzes the effect of modifiers on the rheological characteristics and also considers fatigue resistance with the main aim of preventing rutting in asphalt pavement due to different temperatures. Hence a seminal effort is assessed in evaluation of Portland cement and Waste Animal Bone as cost effective modifying agents. The conventional bitumen penetration grade (80/100) is used in this research. The control specimen four binders were obtained by mixing the asphalt binder with four different percentages (0 %, 1 %, 3 % and 5 %) of PCWAB by weight of asphalt binder. Rheological characteristics were evaluated with Dynamic Shear Rheometer test (DSR) such as Amplitude sweep test (AST), Frequency sweep Test (FST) and Multiple-stress creep and recovery test (MSCR). AST and FST were adopted for determining linear visco-elastic range, Master curve were developed for same temperature (21.1 °C, 37.8 °C, and 54.4 °C) respectively while MSCR and Performance Grade (PG) were determined for four different temperatures (52 °C, 58 °C, 64 °C, and 70 °C). Finally, the master curve shows that 3% and more addition of PCWAB on asphalt binder increases the stiffening property of asphalt binder at high temperatures and low loading frequencies (susceptible for rutting). On the other hand, from the test result obtained from MSCR test the smallest total strain value was 5 % for PCWAB followed by the 3 % and 1 %. Thus, up to 3 % addition of PCWAB significantly improves the resistance of asphalt binder to rutting. In addition, using PCWAB as asphalt binder modifier has a positive impact on environmental preservation. The performance grade determination also depicted the improvement in PG as content of modifier increases. Therefore, the evaluation showed that the 3 % modified binder is better in its rutting performance with improved performance grade.

1. *Introduction*

Road infrastructures have a huge contribution for rapidly economic growth of a country and a world as a whole. Thus rapidly economic growth increases number traffic volume per citizen and heavy axle load [1]. Bitumen binder are organic materials whose binding and hardening properties are caused by the temperature related change of adhesion and cohesion of their molecule [2]. Bitumen is often characterized by composition and the colloidal structure determine its physical and rheological properties [3, 4]. These binders are commonly used in the pavement construction to meet the raising requirements for durability of the road surface. In the road construction industry, asphalt binder used as an intermediate and surface layers of flexible pavement to provide tensile strength which resisting distortion, protect the asphalt binder pavement structure [5, 6]. Rutting in asphalt binder pavements leads to a serious problem that reduction in both structural and also reduce service life of the asphalt pavement [7–10]. Therefore the asphalt pavement roads are used to increases its ability to carry the traffic load under different condition without causing distress [11, 12]. From this perspective significant amount of work has been done using different additives and modifiers. Such as: crumb rubber, Styrene-butadiene-styrene, synthetic resins, metal-organic

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compounds, polyvinyl chloride, ethyl-vinyl-acetate, bone glue, lime, Portland cement, fly ash and paraffin Which improves the physical and rheological properties of asphalt binder [13–15]. Among them, Portland cement and waste animal bone a wide range of viscoelasticity to achieve long-lasting road surface, as it ensures consistency of bitumen rheological state in extreme service temperatures.

Several polymers have been tested as modifier for improving bitumen's performance involving filler, fiber, elastomers and plastomers [16, 17]. The most used modifiers for bitumen performance improvement are polymers. The physical and chemical properties of the polymers modified bitumen are dominated by the properties of the base bitumen [18, 19]. Moreover elastomers (Crumb rubber, styrene-butadiene-styrene and styrene-butadiene rubber) Increase stiffness at higher temperatures, decrease stiffness at lower temperatures to resist thermal cracking and Increase elasticity at medium range temperatures to resist any cracking [16, 20]. And plastomers (polyvinyl chloride, ethyl-vinyl-acetate and ethylene propylene) which increase high temperature performance, structural strength and increase resistance to rutting [19, 21]. As well as filler (lime, Portland cement and fly ash) improves fill voids, increase stability, improve bond between aggregate and binder [22–24] and also researchers investigated on different additives such as nano silica, nano clay as well as fibers and nano charcoal coconut-shell ash as a bitumen modifiers. The study improved the physical and rheological properties of asphalt binder for rutting and fatigue resistance [25, 26]. Additionally; crumb rubber also improve the performance grade of bitumen from PG64 to PG76 for permanent deformation of asphalt at high temperature [27]. 0.75 % soybean- derived biomaterial by weight of bitumen on neat bitumen binder and also polymer modified bitumen showed a significant effect on fatigue properties [28, 29].

Currently the most several researchers have been conducted on bone and cement modification, waste animal bone (WAB) mineral is composed of calcium, phosphate as well as hydroxyl ions, especially cattle bone has ultimate compressive strength and shear strength of more than 140 MPa and 85 MPa respectively. It is viscoelastic materials, with a density of 1810 kg/m^3 and melting point of 1670 °C [30, 31]. Moreover, former researchers conducted on bone glue for modification for enhancing pavement performance without significantly changing the asphalt binder's compaction and mixing temperatures of HMA and viscosity. The results showed that bone glue modification improved complex shear modulus. A modified binders with bone glue showed great improvements on shear fatigue, creep compliance, and complex shear modulus than control asphalt [31]. Furthermore, studies which have been conducted on partial replacement of cement with animal bone in concrete. For instance, a maximum of 10 % bone powder ash was incorporated into cement [32]. The results showed that the replacement of cement with 10 % bone powder ash showed an increase in the compressive strength of concrete. Up to 5 % bone powder replacement of cement increases both compressive and tensile strengths of concrete [33].This is because bone powder is acting as nucleation agent which increases the hydration ability of cement.

Portland cement and waste animal bone the major benefit of this particular combination is synergetic effect. It has been observed for binder elasticity and resistance to permanent deformation [23]. Moreover, Portland cement and waste animal bone improves bitumen stiffness, viscoelastic, to prevent stripping of binder from dried aggregate, increases dry resilient modulus of mixes and Reduce environmental problem. In addition, the stability of binder during long-time storage at elevated temperatures is increased [23, 24] and increases dry resilient modulus of mixes.

Therefore, this study aims to evaluate rheological characteristics and the performance of bitumen modified with Portland cement and Waste Animal Bone at high temperature for rutting resistance. The following binder properties were carried with conventional and Dynamic Shear Rheometer test (DSR) such as penetration, softening point, ductility, Amplitude Sweep Test (AST), Frequency Sweep Test (FST), Performance grade Determination (PG) test and Multiple-Stress Creep and Recovery test (MSCR) for both unaged and aged Rolling thin Film oven (RTFO) Test, as well as for both modified and unmodified bitumen. In developed countries performance-based asphalt binder specifications, super pave binder grading system is in use. Recently the more advanced asphalt binder specification method using MSCR has been conducted. This MSCR test method is becoming popular since it best predicts rutting and convenient to consider different levels of traffic were examined.

2. Material and Methods

This methodology and experimental work focus on identifying the linear viscoelastic (LVE) limit of different unmodified and modified bitumen as a function of different loading times and temperatures. Additionally, upon establishing the LVE limit, the viscoelastic behavior of bitumen in their linear region are evaluated. Understanding the properties of bitumen material is important to quantify its engineering performance. Materials such as bitumen which exhibit aspects of both elastic and viscous behavior, must be characterized with test methods and analytical techniques that account for the time (or rate) of loading and temperature.

The effect of rheological behavior of bitumen improved by modification of bitumen with Portland cement and waste animal bone. As well as every test approaches, equipment and conditioning machines used to investigate asphalt's both conventional and fundamental test Conventional test method such as penetration, ductility and softening test also RTFO (Rolling Thin Film Oven) test has conducted. Fundamental properties are assessed using MARVEL BOHILN Dynamic Shear Rheometer test. The test conducted under dynamic shear rheometer such as AST, FST, MSCR and PG determination. Generally, in this research a comprehensive literature review made to understand the previous efforts, which include the, academic journals, research papers and review of textbooks are conducted in this research.

2.1. Materials

Bitumen binder grade 80/100 penetration was used for the preparation of modified binders blend in this study this bitumen obtaining from chain road construction company and its physical properties penetration softening point, ductility and conditioning test [34–37] Were studied to qualify the bitumen before modification see Table 1. Two different filler additives namely waste animal bone and Portland cement was used for the binder modification. The waste Animal Bone and Portland cement both obtained from local source. According to express on Ethiopian pavement design Manual [10] three different penetration grades of bitumen 40/50, 60/70 and 80/100. For this study selected penetration grade 80/100 which is less stiff and the softer when compared to the others listed in above, the objective of the study, the softer bitumen is selected to evaluate stiffness and resistance against rutting when modified with PC (Portland cement) and WAB (waste Animal bone).

2.2. Sample Preparation in laboratory for modifiers

The first step to clean the Bone samples, can, sun-dried and oven dried to reduce its oil content before Powder bone and screening into the desired sizes. As well as Portland cement is used in this study, the required size of both modifiers to prepare bituminous mastic is (< 75 µm) and their chemical properties also studied and tabulated in Table 2.

Figure 1. Sample preparation for Portland cement and Waste Animal Bone.

2.3. Dynamic shear rheometer DSR test

Dynamic shear Rheometer (DSR) test Used to characterize the rheological behavior of bitumen and rutting resistance at high, intermediate and low Temperature. This was done by measuring the viscous and elastic characteristics of asphalt cement with Standard test for determined the viscoelastic characteristics of asphalt cement using DSR test is described in AASHTO T315-10. First the asphalt binder is heated until sufficient fluid to pour and to prepare the test specimens. Then a small sample of asphalt binder is sandwich between two plates. But before placing the sample the DSR is set to a particular temperature. DSR test utilized to measure different properties of bitumen before modification and after using modification.

The test can be done when the bitumen's sample is sandwiched between two parallel metal plates. The bottom (surface) plate is fixed (constant) and the upper plate oscillate forth and back across the sample to create a shearing action. Depending upon the type of bitumen being tested the test temperature, specimen size and plate diameter varies. Test temperatures greater than 46 °C use a sample 0.04 inches (1 mm) thick and 1 inch (25 mm) in diameter), Test temperatures between 4 °C and 40 °C use a sample 0.08 inches (2 mm) thick and 0.315 (8 mm) in diameter. DSR tests are conducted on BRTFO (Before Rolling Thin Film Oven), ARTFO (After Rolling Thin Film Oven) aged and then the rheological parameter is recorded such as complex shear modulus ($G*$), dynamic viscosity, frequency, phase angle (δ) and accumulated strain, etc. The complex modulus is a determined the resistance of asphalt cement to rutting (deformation) when exposed to a sinusoidal shear stress load and consists both visco-elastics when elastic (recoverable) and viscous (non-recoverable) component of material.

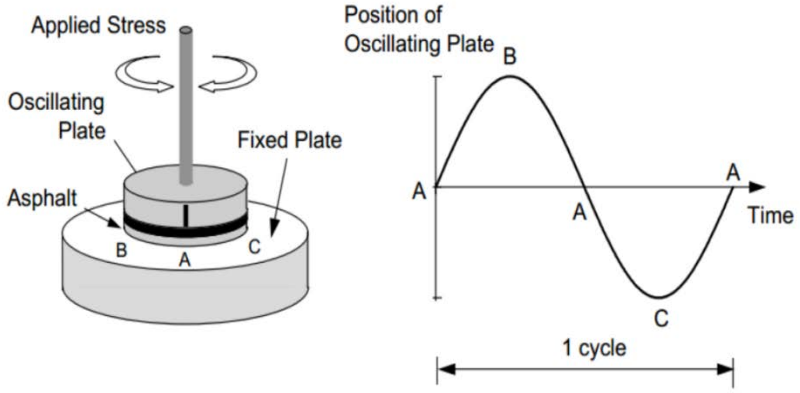

Figure 2**. Working principle of dynamic shear rheometer test [2].**

Phase angle (δ) is an indicator or represent of relative amounts of viscous and elastic components as well as the lag (difference) between the applied (load) shear stress and the resulting get shear strain. The values of complex modules G^* and the value of phase angle (δ) for asphalt binder are dependent on the frequency of loading and test temperature. The operation principle of the DSR is when generalized on the figure as follows. An asphalt sample is sandwiched between an oscillating spindle and the fixed base. As shown in Fig. 2 below the oscillating plate (often called a "spindle") starts at point A and moves to point B. From point B the oscillating plate moves back, passing point A on the way to point C. From point C the plate moves back to point A. This movement, from A to B to C and back to A comprises one cycle [38–40].

There are four main types of tests conducted under DSR test in this research to determine the rheological behavior of bitumen. These are: Amplitude Sweep test, Frequency Sweep Test, Performance grade test and Multiple Stress Creep and Recovery test (MSCR).

2.3.1. Amplitude Sweep Test

The Amplitude sweep test is used to determine the Linear Viscoelastic Range (LVR) of a visco-elastic material. The AST test shear stress is different while frequency kept constant at 1.59 Hz until the sample flow and breaks down and rheological material functions are not independent of the set parameter anymore LVE part is the region where the applied oscillation is nondestructive. In most cases log-log graph on the same scale is plotted as strain in the graph of x-axis and complex modulus G* in the graph of y-axis. The complex shear modulus (G^*) versus x-axis with strain plot used to determine the (LVE) region.

The amplitude sweep test was carried out following the test standard AASHTO T 315 at a constant frequency of 10 rad/sec at specific test temperatures (21.1 °C, 37.8 °C & 54.4 °C). The test was in shear stress control mode with minimum shear stress 100 pa and maximum shear stress 90000 pa.

Prior to mounting the asphalt binder specimen between the plates, the “zero gap” setting must be established at the test temperature because the frame and fixtures in the DSR change dimension with temperature. So, for every 12 °C temperature variation on the test, zero gap was maintained. in order to calculate the strain which is used as an input parameter for frequency sweep test using 8 mm plates and 2 mm gap for low and intermediate temperature (21.1 °C, 37.8 °C) as well as for high temperature 25 mm plate with 1 mm gap are conducted.

2.3.2. Frequency Sweep Test

Frequency sweep test is very important test to evaluate the rheological characteristics of asphalt binder oscillatory test with variable frequency and constant amplitude values. Using this test time dependent shear behavior can be examined. Short-term behavior is simulated by rapid movements (at high frequencies) and long-term behaviors by slow movements (at low frequencies). Parallel plates with diameters of 8 mm and 25 mm were used in the temperature ranges of 21.1 °C, 37.8 °C and 54.4 °C respectively with a frequency range of 0.1 Hz to 25 Hz. The final gap was adjusted to 1 mm for a 25 mm plate and 2 mm gap for the 8 mm spindle. Frequency sweep test helps to evaluate the rheological property of visco-elastic material in the form of complex modulus (G^*) and also phase angle (δ) parameters have been presented as isothermal plots and master curves for each type of bitumen. Because of the large amount of data (result) generated in these tests, these plots were very useful in presenting and interpreting the results. Master curves allow the rheological data to be presented over a wide (high) range of frequencies and temperatures in one plot. Therefore, to avoid presenting a large (huge) number of graphs, the results are mainly presented and analyzed as master curves.

2.3.3. Performance grade determination (PG) test

Performance grade (PG) is a binder specification and mix design procedure developed by Strategic Highway Research Program (SHRP). This asphalt binder specification system works based on climate at which the pavement life is expected to serve by evaluating the contribution or role of the asphalt binder in resistance to permanent deformation (Rutting) at low temperature cracking and also fatigue cracking in asphalt pavements. According to the explanation of super pave, to carry out performance grade determination new set of tests of physical properties at a range of temperatures must be carried out. The performance grade (PG) of the asphalt binder is designated as PGxx-yy, where xx represents the average seven days maximum temperature and yy represents the minimum temperature. Super pave Asphalt Binder Specification the grading system is based on Climate [10]. Performance grade test (PG-test). This test was conducted at high temperature to categorize the tested bitumen binder sample in different temperatures, ± 6 °C increments [21, 41]. For the purpose of this study from the above indicated high temperatures 52 °C, 58 °C, 64 °C, 70 °C and 76 °C are used for performance grade determination and for MSCR test (e.g. PG70 No rutting until 70 °C).

2.3.4. Multiple stress creep and recovery (MSCR) test

MSCR is the latest indicate improvement on Superpave Performance Graded bitumen Binder specifications and addresses or measure at high temperature rutting (permanent deformation) both for unmodified and modified binders. This new test and specification provide that more accurately indicates the rutting performance of the binder and recovery measurement can Identify and quantify how the polymer is working in the binder MSCR is conducted after PG determination test because it relate rutting depth with both the environmental temperature and traffic loading [3, 42]. Sample preparation and the plate are the same as PG test but it require a little software adjustment. A major benefit of the new MSCR test is that it eradicate the need to run tests like as elastic recovery and also phase angle procedures designing in specific to indicate or measure polymers modification of bitumen binder.

Several studies have shown that the $G^*/\sin(\delta)$ based specification does not correlate well with field performance. The test protocol (AASHTO T350) requires that a 25 mm diameter and 1 mm thick asphalt specimen is subjected to 10 cycles of one second creep loading followed by 9 seconds rest period at stress levels of 100 Pa and 3200 Pa at the high PG temperature using a DSR. In this way 20 cycles at the 0.1 kPa stress level followed by 10 cycles at the 3.2 kPa stress level for a total of 30 cycles will be done. The first sample 10 cycle at 0.1 kPa was used for conditioning the specimen. There are no rest periods between creep and recovery cycles or changes in stress level. The total time required for completing the two-step creep and recovery test is 300 s. The sample has to be residue from T 240 (Rolling Thin-Film Oven Test). Generally, in MSCR test a constant stress is assigned and the time-related strain is measured and it is called shear compliance $J(t)$. It defines how complian the specimen is the higher the compliance the easier the sample can be deformed [38, 43]. From the MSCR test we can determine the following main parameters:

i. Non-recoverable creep compliance;
ii. Percent difference between non-recoverable creep compliance;
iii. Average percent recovery;
iv. Percent difference in recovery.

3. Result and Discussion

The test results and analysis have been organized under result and discussion. The result and discussion divide into two main parts: (I) Analysis of conventional characteristic of asphalt binder and (II) Analysis of rheological characteristic of asphalt binder. It starts with test parameter setup and trial tests. This part helped to acquire adequate experience on how to conduct the tests in a better way. The test procedure, specifications and methodology were described before. Generally, this result and discussion covers every test approaches, equipment and conditioning machines used to investigate binder containing different proportion of Portland cement and waste animal bone powder for both conventional and rheological properties. Rheological properties are assessed using MARVEL BOHILN Dynamic Shear Rheometer test. A summary of the test results is presented in Fig. 1–11 and Tables 1–4 have been organized to evaluate the binder characteristics in the following sections.

3.1. Elementary Analysis of Portland cement and waste Animal bone

As a result of the other researchers conducted original bitumen is mostly affected by chemical, physical and rheological characteristics of bitumen [39]. Rheological properties of asphalts with different contents of nano-silica and rock asphalt were analyzed by univariate analysis and variance analysis [26]. The results of DSR test revealed that Qingcheng (QC) rock asphalt had a remarkable impact on the complex shear modulus (G) and phase angle, while the effects of nano-silica were relatively small, which mainly improved complex modules (G) [25]. As well as filler (lime, Portland cement and fly ash) improves fill voids, increase stability, improve bond between aggregate and binder [23–25], the result obtained from chemical component of Portland cement and waste animal bone test are displayed in Table 1. The result was conducted in Ethiopian central geological survey laboratory. Chemical component of Portland cement and waste animal bone varies depend on type of cement and bone. The raw materials found in the manufacture of Portland cement and waste animal bone consist mainly of lime, silicon, alumina and iron oxide (CaO, SiO_2, Al_2O_3, and Fe_2O_3) [24]. Table 1 below shows the chemical analysis of Portland cement and waste animal bone.

Table 1. Chemical analysis of Portland cement and waste animal bone.

Chemical	Animal bone (%)	Cement (%)
Sio_2	5.28	43.62
Al_2O_3	0.01	3.92
Fe_2O_3	0.40	2.92
Cao	42.92	43.80
MgO	0.94	1.00
Na_2o	1.34	0.32
K_2O	0.30	0.20
MnO	0.02	0.12
P_2O_5	26.94	0.14
TiO_2	0.01	0.25
H_2O	2.24	0.80
LOI	18.80	3.212

From the Table 1 above it is observed high percent of chemical component found in the manufacture of Portland cement and waste animal bone consist of calcium oxide (Cao). High percent of calcium oxide (Cao) implies high resilience modules, high softening point, High resist of moisture damage and hardest material [24] as well as silicon dioxide (Sio_2) and alumina oxide Al_2O_3 high role to improved pavement performance in Road construction [12].

3.2. Conventional test method results

The bitumen has a limited range of rheological properties and durability that are not sufficient to resist pavement distress [15]. Penetration grade 80/100 neat bitumen selected for modification since it is softer, low stiffness and not used for high temperature and became more viscous at high temperature [34–37]. Therefore, depend on above reason the quality of original bitumen was checked for its conformity by conducting conventional tests. And summary of convention test results with specification requirement are presented in Table 2 below.

Table 2. Summary of Convention Test Results with specification requirement.

Test type	Unaged	aged	Spec.(unaged)	Spec.(aged)
Penetration	97	77	80-100	50 (Min)
Ductility	100+	100+	100 (Min)	75 (Min)
Softening point	46		42-51	75 (Min)
RTFO		0.05%		0.8 (Max)

The conventional test result of the original bitumen was checked for its conformity with its specification requirement. As shown in Table 2 above the binder qualifies to be modified and to be evaluated with further fundamental tests as a control material.

3.3. Amplitude sweep test Result

Stiffness of asphalt binder and testing temperature had a significant effect on linear viscoelastic range [44]. The result obtained from amplitude sweep test, linear viscoelastic range, used for as an input for frequency sweep test. Thus, asphalt binder is viscoelastic material which expressed as a function of time and temperature. The linear viscoelastic range (LVR) illustrated in Fig. 3 and 4 by the relationship between the complex shear modulus G* versus with shear strain. In SHRP study it is reported the linear viscoelastic range (LVR) was defined as the point where complex modulus decrease to 95 % of its initial value [17]. As a result of the other researchers conducted a modified binder with bone glue showed great improvements on shear fatigue, creep compliance, and complex shear modulus than control asphalt [31]. Based on this amplitude sweep test result from Fig. 3 and 4 indicated Portland cement and waste animal bone improves complex modules and linear viscoelastic behavior at high, intermediate and low temperature. limits, fundamental tests used the DSR test was performed at stress level well inside the linear region and the linear fundamental characters for modified and original bitumen. AST provide LVE-range by limiting strain value [7]. So that asphalt binder for which the stress-strain behavior is linear and independent of rate of loading and temperature Table 3 below illustrate linear viscoelastic range limiting strain value. Fig. 3 and 4 show (G^*) and (δ) versus temperature, obtained from DSR test of base and PCWAB modified asphalt binder before and after RTFO aging respectively. As shown in the Fig. 3 modification of Portland cement and waste animal bone improves linear viscoelastic also has shown in the Fig. 4 that the stiffness of asphalt binder decrease as the test temperature increases and the LVE-range becoming wider than lower test temperature at 3 % PCWAB addition. Fig. 3 expresses linear visco-elastic range for 3 % modified binder after aged.

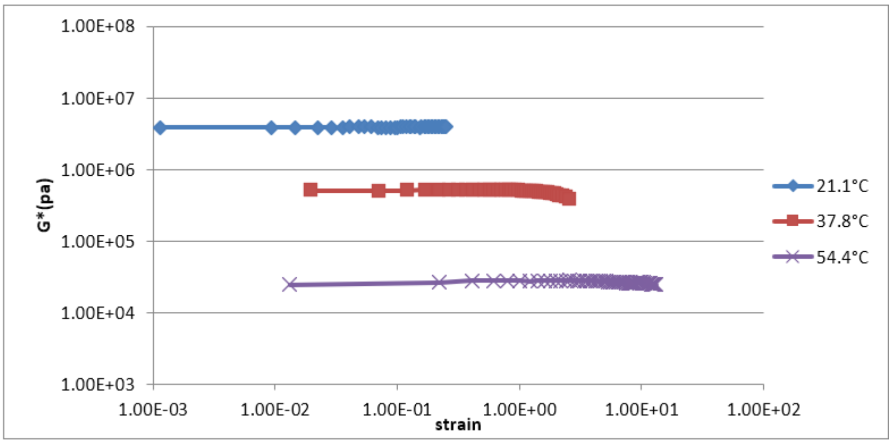

Figure 3. Linear visco-elastic range for 3% modified binder after aged.

Generally, in this study the required output from amplitude sweep test is determining the linear viscoelastic range of all binder containing different proportion of PCWAB at each temperature and in both unaged and RTFO aged conditions. Moreover, Portland cement and waste animal bone improves bitumen stiffness, viscoelastic, to prevent stripping of binder from dried aggregate, increases dry resilient modulus of mixes and reduce environmental problem.

Based on the results shown below in Table 3 and Fig. 4, the temperature, percent proportion of PCWAB and aging have a significant effect on the LVE-region. So, highest content of PCWAB lower linear visco-elastic range and higher stiffness. Fig. 4. below shows linear visco-elastic range for 3 % modified binder before aged.

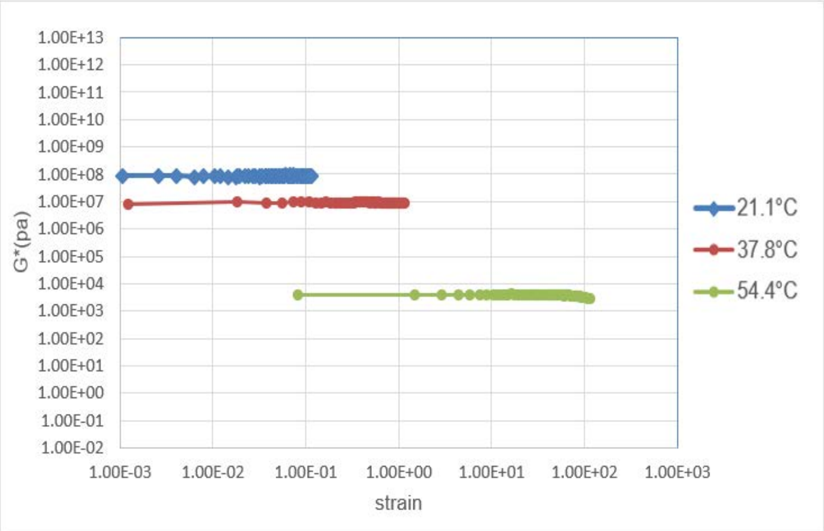

Figure 1. Linear visco-elastic Range for 3% modified binder before aged.

Based on the test result represented graphically the limiting strain values were analyzed considering the plateau strain values of each sample. The plateau modulus values are almost a constant value of $G*$ is observed for different values of shear stress and shear strain before the graph declines [19]. In this study the LVE strain limit can be calculated as the point beyond which the measured value of $G*$ decreased to 95 % of its zero-strain as shown on above Fig. 3 and 4. Finally for each and every curves of amplitude sweep results, a horizontal line along 0.95$G*$ will be constructed to intersect the curve at a point. Then the corresponding strain value of that intersection point will be considered to be the limiting stain value (γL). Table 3. Shows below Summary of linear viscoelastic limiting strain range value.

Table 3. Summary of linear viscoelastic range value.

Temperature	Modified (%)	0.95$G*$	Limiting strain	ARTFO
21.1°C	0	8.43E+05	3.10	2.44
	1	2.42E+06	2.58	1.10
	3	3.75E+06	2.05	1.06
	5	7.38E+05	1.02	1.01
54.4°C	0	3.04E+03	74.08	53.01
	1	3.83E+03	58.94	48.19
	3	3.54E+03	49.90	36
	5	3.81E+03	31.00	32.85

The results obtained by other authors the response of LVE affected due to temperature, content of modifier and aging [27]. Depend on the summary Table 3 above the stiffness of asphalt binder increases with addition of PC and WAB. Binder with 5 % PCWAB have highest stiffness and lowest LVE-range at constant temperature. Conversely the lower stiffness shows a larger LVE-range; indicating it is more flexible and has better performance for the lower temperature regions. At 21.1 °C temperature the linear visco-elastic value for each sample is minimum and the effect of the modifier is not significant. At 54.4 °C temperature higher linear visco-elastic values are observed as the material gets softer or less stiff.

3.4. Frequency Sweep Test Result

Frequency sweep test (FST) data results are manipulated mainly used for constructing Master curve. Isothermal plots with log-log scale for complex shear modulus. The frequency varies from 25 Hz to 0.1 Hz to represent the damaging effect of the traffic load. The major rheological parameters determined from the FST plots to make some rheological discussions, but it is essential to make the observation and discussion after developing of master curves [45].

Isothermal plots -this plots the complex shear modulus as a function of frequency for all binders were represented Fig. 5 below shows Isothermal plots for 3 % binder Modified before aged isothermal graphs considering temperature, complex shear modulus, frequency, ageing and content of modifiers [21]. Isothermal Plots as shows on above Fig. 5 and 6 that by simply looking as the temperature increases the shear stiffness decreases for all the binders. The increase in content of modifier increases with complex modulus and also aging improves shear stiffness of the binder (complex modulus).

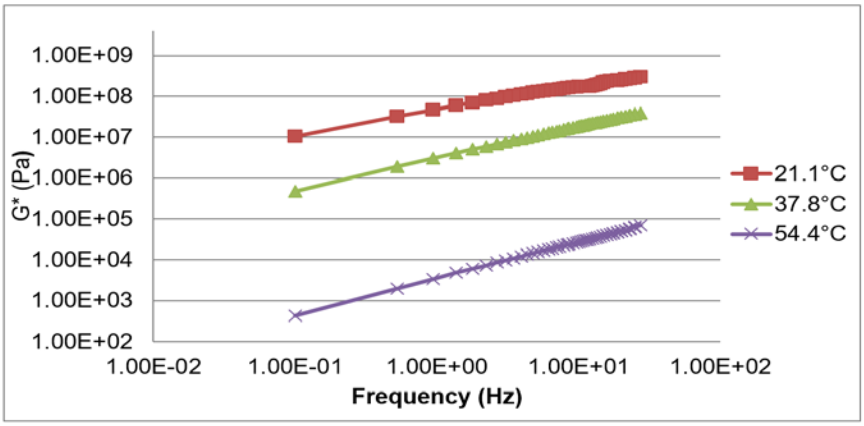

Figure 2. Isothermal plots 3% Modified binder before aged.

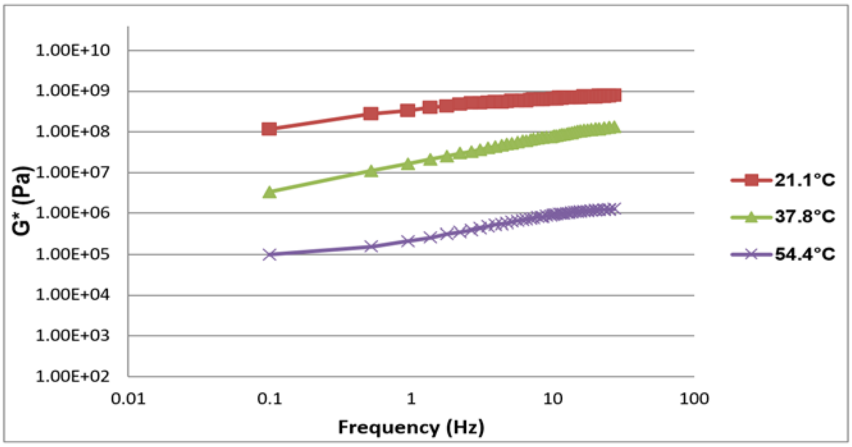

Figure 3. Isothermal plots (3 % binder Modified After aged.

3.5. Master Curves of Complex Shear Modulus

The construction of master curves is a powerful tool to understand the rheological properties of asphalt binder. The data at any other temperatures were shifted with respect to time until various curves overlap almost perfectly to form a single master curve using time-temperature superposition principles. Binder master curve graph extrapolate the test result up to very high frequency and very low frequency [46]. Also at high temperatures the modified bitumen binder has high complex shear modulus which is taken as indicate stiffness that helps for resisting rutting [16, 41]. It can be possible to seen that the addition of modifiers leads to a bitumen binder which performs good or better under high temperatures and also low frequencies (lower values of phase angle in pascal) which compared with neat bitumen shows Fig. 8 and 9.

At higher frequency values or low temperatures the ratio of increment of phase angle values due to modification becomes small, modulus master curves were developed using microsoft excel solver to best fit the sigmoid function for all asphalt binders [44]. The sigmoid function to best fit the obtained G^* data from the DSR is carried out with trial and error by changing the sigmoid constants and the shift factor. The sigmoid function to calculate the complex shear modulus is represented as. Different scholars use different models for shifting to single reference temperatures. But a research developed at the University of Maryland showed that the master curve for binders which represented by a sigmoidal function defined by equation.

$$log\left|G\right| = a + \frac{b}{1 + r^{d\,log\,fr + \gamma}} \quad (1)$$

where a, b, c and d are sigmoid function constants.

fr is reduced frequency.

$|G^*|$ is complex shear modulus

In this research the master curves is constructed fitting a sigmoidal function to the measured the complex modulus test data which can be done by solver function in the excel spreadsheet and minimizing the error between the predicted value and the obtained value of the complex modulus at reference temperature 21.1 °C. William-Landel-Ferry (WLF) empirical relationship shown below was proposed by Williams to link the shift factor for each flow curve to the master curve, based on the time-temperature superposition to obtain the shift factor (aT) [47].

$$log\,aT = \frac{-c_1(T - T_{ref})}{c_2 + (T - T_{ref})} \quad (2)$$

where T is temperature, T_{ref} is the reference temperature c_1 and c_2 are taken as constants. From DSR data result three test temperatures (21.1 °C, 37.8 °C, and 54.4 °C) were used to construct the master curves for complex modules and the shift factors have been developed to construct the master curves for aged and unaged binder. All of these values vary for each aged and unaged binder. As for the temperature shift factors, a21.1 is zero for all the binder types because all the parameters are shifted to 21.1 °C. Whereas for a37.8 and a54.4 the values are all negative because the stiffness parameters are shifted to reduced temperature which is 21.1 °C.

Before developed final master curve, complex modules as a function of reduced frequency is plotted to illustrate how the temperature shift factors were used see in Fig. 7 below.

Reduced frequency is determined as;

$$log\ fr = log_f\ p + aT \tag{3}$$

where fr is reduced frequency, f is frequency and aT is temperature shift factor.

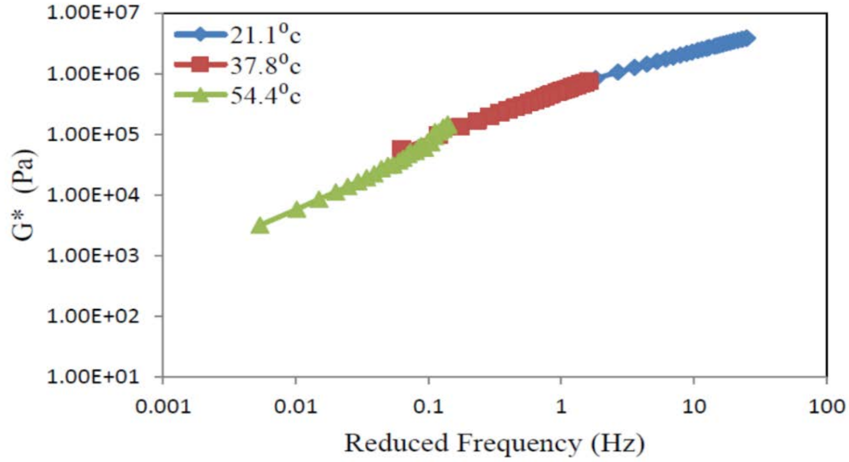

Figure 7. Temperature Shift @21.1°C Ref. Temperature for 0 % modified after RTFO.

Finally, modulus master curves were developed for both after modified and before modified binder Fig. 8 and 9 below shows Complex modulus master curve before aged and after aged respectively.

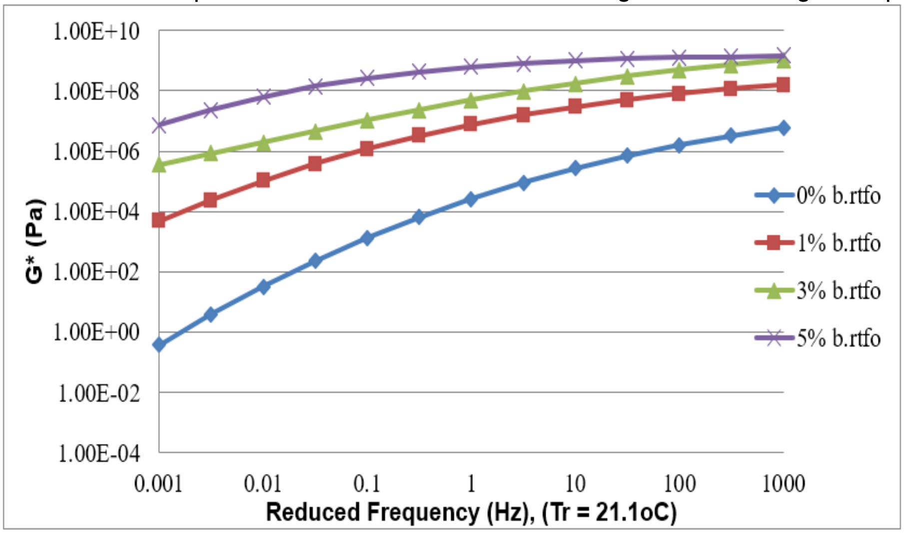

Figure 8. Complex modulus master curve before aged.

Furthermore, studies which have been conducted on partial replacement of cement with animal bone in concrete. For instance, a maximum of 10 % bone powder ash was incorporated into cement [32]. The results showed that the replacement of cement with 10 % bone powder ash showed an increase in the compressive strength of concrete. Up to 5 % bone powder replacement of cement increases both compressive and tensile strengths of concrete [33].This is because bone powder is acting as nucleation agent which increases the hydration ability of cement. As shown on the above Fig. 8 and below Fig. 9, it can be seen that stiffness of PCWAB increases from 1 % to 5% for unaged and aged bitumen at high temperature and low loading frequency this indicate modifier has great role for resist permanent deformation (rutting) at high Temperature. Addition of Portland cement and waste animal bone (PCWAB) has a positive effect on asphalt binder thereby increasing its stiffness and when temperature increasing also shear stiffness decreases. This implies that at high temperature and low frequency the smaller value of shear modulus is pronounced. Fig. 9 shows Complex modulus master curve after aged.

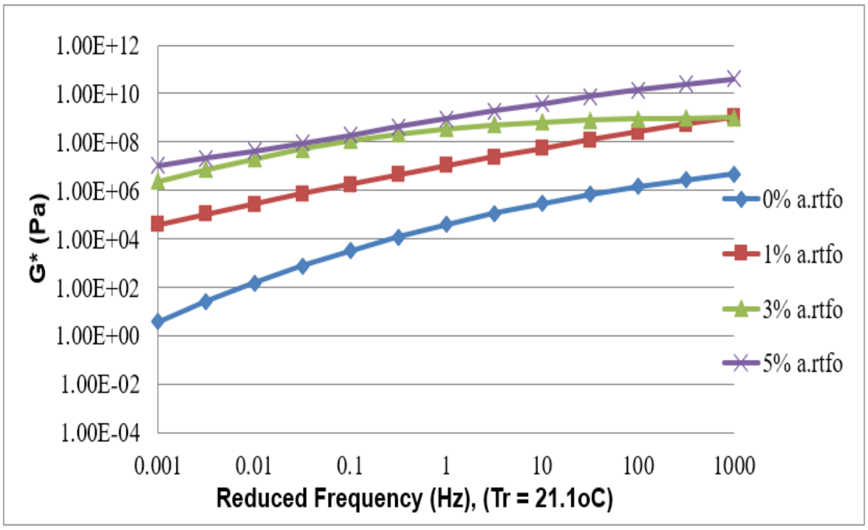

Figure 4. Complex modulus master curve after aged.

Permanent deformation (rutting) is a serious problem at high temperature due to slow moving traffic and as observed the as addition of modifier improves the pavement performance and decreasing the problem of distress by increasing the stiffness of the binder [41]. According to show on the above Fig. 9 addition of PCWAB has an advantage thereby improving the stiffness of asphalt binder for all loading frequencies and temperature conditions. But addition of 3 percent (%) proportion of PCWAB has no effect at low, intermediate and high temperature. At low frequency and high temperature, the modulus increases appreciably as modifier increases finally when we can summarize that the modifier appreciably improves the complex shear modulus of the original bitumen at low, intermediate and higher temperatures.

3.6. Performance Grade (PG) Determination

PG determination test is conducted at high temperature. As result shows crumb rubber also improve the performance grade of bitumen from PG64 to PG76 for permanent deformation of asphalt at high temperature [48]. As well as PG-temperature grade of base asphalt was shifted from PG58-xx to PG76-xx for PMN binders with concentrations of 7 % by weight. The PG76-xx binder can be successfully implemented into pavement construction in hot climates, owing to its good high-temperature performance [49]. The final rheological parameter $G^*/\sin\delta$, was believed to indicate the rutting resistance at high temperature of asphalt binder before MSCR test was discovered [27]. Performance grade of Portland cement and waste animal bone improve PG 58 to PG 70. The increment in PG clearly shows that modifying bitumen with this PCWAB material increases the stiffness of the asphalt binder. This study uses the parameter $G^*/\sin\delta$ to identify the maximum temperature that the asphalt binder could meet the minimum criteria of AASHTO M-320. The high temperature test result (output from the software) were organized in Tables 4 below illustrated for both before aged and after RTFO (Rolling Thin Film Oven) respectively.

Table 4. Determined High Temperature Performance Grade (AASHTO M 320).

Description	PG (brtfo)	PG (artfo)	PG(HT)
0% modified	58	58	58
1% modified	58	58	58
3% modified	64	70	64
5% modified	64	70	64

From the Table 4 above it is observed that there is a PG grade improvement from PG 58 to PG 64 when the neat bitumen is modified with 3 % PCWAB. When the content of the modifier is increased to 5 % then again, the PG will be further improved to PG70. Finally; from above we can summarize that the modifier appreciably improves the complex shear modulus of the virgin binder at higher temperatures and PG asphalt binder rutting resistance parameter $G^*/\sin\delta$ is based on complex modulus and phase angle. As mentioned above it has its own shortcomings to properly simulate field rutting performance. Based on this it is possible to guess that the improvement in rutting performance was pronounced due to the modifier. But recently it is proven that the current performance grade stiffness parameter ($G^*/\sin\delta$) is weak in predicting rutting potential [8]. Therefore; it is better to further evaluate the modified binder by MSCR tests since the parameter from this test termed as non-recoverable creep compliance best relates with rutting than any other parameter from binder test.

3.7. Multiple Stress Creep and Recovery (MCSR) Test Results

Fig. 10 displays the accumulated strain of PCWAB modified asphalt. This test is performed using the Dynamic Shear Rheometer test (DSR) by applying a controlled shear stress (100 and 3200 Pa) using a load for 1 second followed with 9-second rest period. During each cycle time the bitumen binder reaches a maximum or peak strain and also recovers before the next cycle stress is also applied again. The permanent strain is then accumulated for 10 cycle's totals of 100 seconds. This implies it is good information regarding the rheological characteristic of asphalt binder [3, 6]. Comparing Fig. 10 and 11 below it can be realized that a higher creep stress level was associated with a higher accumulated creep strain levels; it means that the stress level has a serious effect on the accumulated strain and the growth rate of strain direct proportion with stress level.

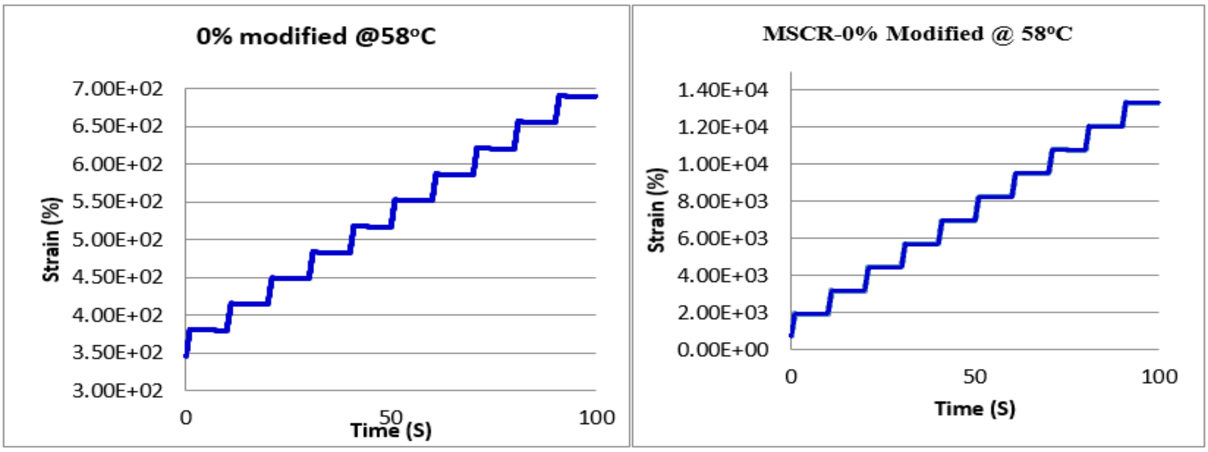

Figure 10. MSCR result of 0 % modified bitumen at 0.1 kPa and 3.2 kPa respectively.

Furthermore, Multiple Stress Creep Recovery (MSCR) tests conducted at a pre-determined temperature increasing the bitumen viscosity by blending in a micro-sized calcium carbonate filler or a nano-sized organo-clay filler were shown to affect the stress sensitivity but not the actual magnitude of failure stress which remained in line with the base bitumen. On the other hand, the SBS-modified bitumen had completely altered shear stress failure criterion. When considering non-polymer-modified bitumen's, simple high temperature strain sweeps have also been shown to be a potentially rapid and very useful creep performance ranking tool [9].

The MSCR test result (software output) contains huge data to represent in tables here. The Multiple stress creep and recovery test demonstrate that the PCWAB dramatically increases the resistance of binder to permanent deformation. In order to evaluate the strain response by asphalt binder to stress, the non-recoverable compliance (Jnr) is presented in Fig. 11 and Table 5 below. The Jnr is generally used as indicating to resistance against deformation of the binder at high temperature and repeated loading [50]. From above Fig. 10 Jnr values at both stress level (0.1 kPa and 3.2 kPa) of unmodified asphalt are greater than that of PCWAB modified asphalt at all temperature range. The Jnr decreased from 3.88 to 2.28 kPa-1 for as the dosage of PCWAB increased from 0 % to 5 %. This implies, the addition of PCWAB decreased the non-recoverable deformation of binder and can contribute to the rutting resistance of associated asphalt mixtures.

Table 5. MSCR test Summaries of Analyzed Jnr and % Recovery for Modified Binder.

Modified, %	0%	1%	3%	5%
Temperature °C	58	58	70	70
% Recovery @0.1kpa	5.32	26.33	16.82	10.03
% Recovery @3.2kpa	0.84	4.38	2.67	1.69
% Recovery difference	84.28	83.34	84.14	83.13
Jnr@0.1kpa	3.38	2.35	2.56	2.72
Jnr@3.2kpa	3.88	3.76	2.67	2.28
Jnr difference, %	14.76	60.15	31.01	20.66

Based on above result it is possible to evaluate all the binders by contrasting the calculated basic MSCR parameters with respect to the limits of those parameters described under standard specification for PG asphalt binder using MSCR test, AASHTO M 322. Percent difference in non-recoverable creep compliance between 0.1 kPa and 3.2 kPa is also a requirement with maximum value 75 and Jnr indicated the Rutting resistance at high temperature and repeated load [3, 42, 51]. Therefore, MSCR test Result concluded as follows.

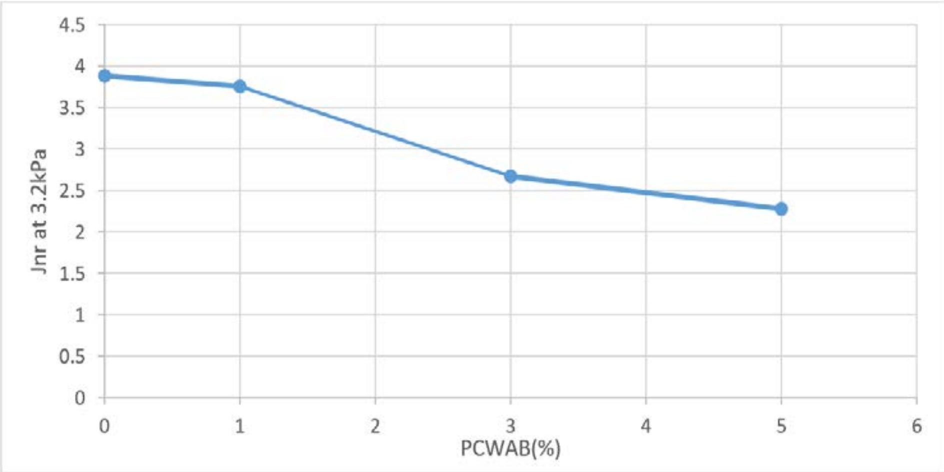

Figure 11. Effect of Portland cement and waste animal bone content on MSCR.

- The Jnr at 3.2 kPa is the fundamental parameter to evaluate rutting potential. The neat bitumen (0% modified) and the 1 % PCWAB modified binder have similar test temperature but different Jnr results. These two binders with test temperature 52, 58 & 64 °C will not be used for heavy and extremely heavy traffic in all cases of their test temperatures.
- The 3 % modified binder with test temperatures 58, 64 & 70 °C can be used for simply heavy traffic at 64 and for standard traffic at 70 °C. This binder shows a significant improvement than the previous two binders in rutting resistance by enhancing up the neat bitumen by one PG grade as per MSCR test also.
- A very good Jnr result is observed for 5 % modified binder this implies as content of modifier increasing Jnr give best result.
- To prove the improvement in rut performance, it is possible to make a clear comparison of Jnr values at the same test temperature (58 and 70). At this temperature the Jnr values are 3.88 and 2.28 for 0 % and 5 % modified binders respectively, which is a difference in rut resistance as per AASHTO M 332.

4. Conclusion

The main objectives of this research were to assessing the rheological characterize of locally available material with asphalt binders and the performance of bitumen modified with PC and WAB at high temperature in rutting resistance and at intermediate temperature in fatigue cracking resistance Using DSR test, this modifier is a new material mixed with a content of 1 %, 3 %, and 5 % percent by weight of binder was mixed with 80/100 penetration grade bitumen. Also, several laboratory tests are conducted in this research. After conducting laboratory tests on asphalt binder and analyzing the data, the results shows that the addition of modifier to the pure bitumen change the rheological characteristic of bitumen and its improved rheological behavior of the bitumen.

Depend on the laboratory results obtained from this study following conclusions have been drawn

1. Dynamic shear rheometer test rheological analysis shows that addition of PCWAB improves the viscoelastic rheological properties of the modified binders at high, intermediate and low intermediate.

2. Estimations of aging and rutting deformation indicates that, the modified binders will have significantly higher resistance to rutting (permanent deformation and aging has less effect on the modified binders as compared to control unmodified binder.

3. From test result obtained from FST, the master curve shows an improving behavior for asphalt binder up on addition 3 % and more percent addition of PCWAB on asphalt binder increases the stiffness property of asphalt binder at low loading frequencies and high temperature (rutting is susceptible). For all binders in almost similar pattern shear stiffness decreases as temperature increases.

4. At low frequency (such as from 0.01 up to 0.0001 Hz or below) and high temperature the effect of content of modifier is observed with difference in larger values of modulus. From this we can summarize that the modifier appreciably improves the modulus of the original bitumen at higher temperature and low frequency.

5. PG grade improvement from PG 58 °C to PG 64 °C. Was observed when the original bitumen is modified with 3 % PCWAB and when modified conducted with 5 % it was increased to PG 70 °C. This indicated as percent content of PCWAB increases also complex shear modulus (G^*) is increasing.

6. From the test result obtained from MSCR, the smallest total strain value was obtained for PCWAB of 5 %, followed by the 3 %, and 1 % PCWAB therefore addition of PCWAB improves the resistance of asphalt pavements to rutting.

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