CREEP AND MORPHOLOGICAL EVALUATION OF POLYPROPYLENE WASTE MODIFIED ASPHALT FOR PAVEMENT APPLICATIONS

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ABSTRACT
Synoptic findings by researchers have revealed tremendous physic-chemical improvements of polymer modified mixes over the conventional asphalt. Traditionally, laboratory mechanical properties were carried out for asphalt testing, but cannot calibrate simple performance test (SPTs) criteria for fatigue and field performance. Marshall test-sized specimens of polymer asphalt mixtures were engineered with arbitrary contents of 0 to 3.0% polypropylene waste admixed with 4.5 to 6.5% bitumen contents based on relevant literature. Creep deformation involves uniaxial static creep (USC) test using BS 598-111. Morphological examinations were test with Hitachi S-4700 field-emission scan-electron-microscope (FE-SEM). Thirdly, thermal degradation was determined using Shimadzu TGA-50 thermo-gravimetric analyzer. The results showed creep resistivity with fatigue recovery of 23.2% and 28.9% strain reduction at 10ºC and 60ºC respectively from the optimal 2.0% polypropylene and 6.0% bitumen compared to the control mix. Also, the same mix produced well dispersed and better enhanced pore packaging micro-structure capable of resisting ageing volatization under severe traffic and environmental loading conditions considered.

Keywords: Asphalt pavement, polypropylene, creep deformation, age volatization and microstructure

1. INTRODUCTION
Increasing traffic loading and exogenous factors have led to many pavement distresses and premature failures. It is important to study some of the causal factors to avert or mitigate the failures. Bitumen ageing, for instance, causes physical, chemical, structural and rheological change of properties of the material and often times, leads to loss of serviceability and functionality of pavement structures [1]. Asphalt creep is a simple performance test (SPT) criterion for evaluating ageing process which often leads to morphological changes and loss of durability [2]. Ageing sensitivity in asphalt mixtures could be due to changes in physic-chemical phenomenon which includes loading stress [3], temperature [4], frequency of exposure or time [5], ultra violet irradiation [6], gamma and microwave rays [7].

Researchers have found that negative effects of free acids and monovalent salts on asphalt and presence of moisture on asphalt reduces both chemical and mechanical bonds in asphalt matrix and could lead to disintegration [8- 10]. Also, oxidation of asphaltenes and resins have been found to alter consistency properties including viscosity, penetration index, and softening point [11] and viscoelastic properties [12; 13].

Asphalt creep is a gradual, but time, traffic and environment dependent deformation and the physical science for study of flow and deformation is called rheology. Previous works on styrene butadiene styrene (SBS) and crumb rubber modified bitumen were reported to have exhibited self-healing during shear and were discovered to produce good rheological and ageing resistant properties [14, 15]. It

has been found that many polymer materials including poly-ethylene, polyester, Poly styrene-butadiene-styrene (SBS), poly vinyl chloride, could effectively increase rutting resistance and bleeding resistance at high temperatures as well as fatigue resistance at intermediate temperatures [5, 15, 16, 20, 36]. This research studies the creep and morphological properties of polypropylene waste modified asphalt for mitigating pavement failures.

2. LITERATURE REVIEW
Asphalt mixes are calibrated by the characteristics of their physical, chemical and rheological properties relevant to mitigating distresses which threatens durability, service and life-cycle problems of pavement. Many aspects of pavement properties have been studied to improve on the rheology and deformation [17]. Studies by [18] on creep deformation and ageing process concluded that physic-chemical the processes leading to failure could be improved by the use of nano-silica admixtures. Creep is defined as the slow time dependent deformation of a material measured under a constant or sustained loading stress [19].

The rate of creep deformation is a function of properties of material, exposure time, exposure temperature and the structural load applied [20, 21]. In evaluation of linear viscoelastic behaviour of HMA materials, researchers generally agreed that creep may be recoverable over time after shear stress relief if permanent deformation is not reached [22-24]. Based on standard protocols, static axial stresses of values between 100 — 206.9 kPa are usually applied as depictions and threshold measures of resilience to standardized field pavement tyre pressures which is as high as 828 kPa under ambient temperature condition in excess of 60°C (140°F) [16, 19, 36].

Asphalt aging is a physic-chemical process contra indicated against temperature susceptibility and volatilization which can affect the volume, entropy and enthalpy of material [25-29]. The mechanical response of asphalt is viscous at high temperatures and glassy or brittle at low-temperatures [24]. In terms of rheological properties, it follows that the mix may become stiffer, inelastic, and more brittle after ageing. Also, the volumetric properties, dynamic modulus due to load deformation and climatic durability of the field testing conditions are important [30-36]. O'Flahery [35] concluded that shear deformations occurring as a result of high shear stresses in the top portion of a bituminous layer may

be considered to be the primary cause of rutting in flexible pavements and a product of creep by gradual low stress level induced by mechanical or temperature loading. But [16, 37] gave an empirical findings that simulates tertiary creep between 414—500kPa stress values.

In order to understand the process of bitumen ageing which often manifest in pavement distresses, durability and other life-cycle problems, it is important to study both mechanical and chemical properties indicated against ageing. Relevant tests conducted for study includes creep test, Scan electron microscopy (SEM), differential scanning calorimetry (DSC) and dynamic mechanical spectroscopy (DMS) [17]. Asphalt ageing induces hardening and increase in viscosity of bitumen which suffers natural degradation by environmental effects [45].

In the morphology, shape and properties of asphalt, [46] noticed some structural and chemical transformations brought by admixing SBS Triblock Copolymer in asphalt. These transformations are favourable to ageing, fatigue and deformation resistances. It was observed therefore, that the morphological changes and ageing resistance are complimentary properties which impacts on the degradation chemistry, packing structure, pore sizes, fibre distribution and physical dispersion of the admixed materials [6- 8, 18]. The physico-chemical transformation and change of phase as a result of physical and environmental factors may be assessed using thermo-gravimetric analysis (TGA), Differential Thermal Analysis (DTA) and Fourier transform infrared spectroscopy (FTIR) [18].

3. METHODOLOGY
3.1 Development of the sample mixes
The development of the mix followed Marshall test procedures outlined by Asphalt Institute (1997) to compact standard specimens weighing 1200g were compacted with 75 hammer blows on each side in standard dimension moulds (101.5mm diameter and 63.5mm height) to simulate heavy traffic situation of greater than 10^6 ESALs. These sample specimens were used to carry out creep test. For morphological and ageing test, the Marshall test optimum bitumen content recorded at 6.0% total weight were sampled and thoroughly mixed with 0, 1, 2 and 3% weight of polypropylene to be within the maximum range of 5% polymer content recommended by [69] in his broad based review of polymer applications in bituminous mixtures.

3.2 Creep Deformation
3.2.1 Short-term Loading (Uniaxial Static Creep Test)

Creep test was performed at temperatures ranging from 10 to 60°C according to the recommendations of BS 598-111: [47]. Marshall specimens were pre-conditioned to testing temperature; and a static load of 414 kPa (60 psi) was applied; being the realistic stress state which correlates with field conditions [17]. The load was applied for 1 hour and the sample was allowed to recover for another hour. Triplicate samples were tested at 10, 25, 40 and 60°C and the cumulative strains were determined at time intervals of 0.1, 0.25, 0.5, 1, 2, 4, 8, 15, 30, 45, 60mins.

In static creep, a loading plate is usually mounted on top of each test specimen and the linearly variable differential transducers (LVDT's) are mounted against the loading plate at points intervals equal to 1/3 the circumference of the plate for recording values and averaging them. At low stress level, a correction factor from repeated creep test multiplied by creep test strain gives the expected rut depth (Static loading and unloading may be applied to each specimen in all duration of two hours (one hour loading and one hour unloading) for either four or six inch diameter.

While conducting the test, axial deformation is continuously observed with respect to time. If the initial height of the specimen and the axial strain, are known; the stiffness modulus S_{mix}, can be determined at any loading time using Equations 1 to 2 given by Al-Qadi *et al.* [68]

$$S_{mix} = \gamma / \varepsilon \qquad (1)$$

$$\varepsilon = h / H_0 \qquad (2)$$

Where γ = Applied stress (N/mm^2); ε = Accumulated strain;
h = Axial deformation (mm); H_0 = Initial specimen height (mm)

3.2.2 Long-term Loading (Uniaxial Static Creep Test)

Also, the test followed recommendations of BS 598-111: [47]. It was conducted at 25°C at the same loading stress of 414kpa [17] and it lasted for 200days to assess tertiary creep that gives rise to rutting.

3.3 Thermal Gravimetric Analysis

The TGA curves and its differential (DTG) were carried out in a Shimadzu TGA-50 thermo-gravimetric analyzer. Ozawa method was used to the apparent activation energy which is a function of degree of decomposition in air and nitrogen gas. Thermal decomposition was determined using 30 mg bitumen samples in aluminium holder under a nitrogen or air flow (50 cm^3/ min), heated from 25 to 630 °C at varying heating rates of 5, 10, 20 and 40 °C/min.

TGA is a material weight loss as a result of degradation under temperature with time [38]; [39]. Apart from degradation mechanism studies, it is used in prediction measurement of service lifetime of materials. DTA monitors the temperature difference existing between a sample and a reference material as a function of time and/or temperature assuming that both sample and reference are subjected to the same environment at a selected heating or cooling rate [40]. The plot of ΔT as a function of temperature is termed a DTA curve; endothermic transitions are plotted downward on the *y*-axis, while temperature (or time) is plotted on the *x*-axis.

The minimal and maximal temperatures of accelerated aging are within range of chemical stability where no chemical changes are detected in a material [41]. At the accelerated temperatures, changes in mechanical properties (elongation at breaking point and traction resistance) and the thermal responses are exhibited by a first order phase change (melting or softening); followed by exothermic change and thermo-oxidative degradation [5, 42]. The oxidative ageing of bitumen gives rise to functional groups including carboxylic acids, ketones, sulfoxides and anhydrides [43].

The range of chemical stability, as well as the temperatures corresponding to the phase transitions can be determined by thermal analysis methods - Thermogravimetric Analysis (TGA), Differential Thermal Gravimetry or Analysis (DTG/DTA) and Differential Scan Calorimetry (DSC) [44]. Weight loss from TG/DTA machine recorded by computer is plotted as a function of time for isothermal studies and as a function of temperature for experiments at constant heating rate. According to [40], degradation or service lifetime prediction is calculated by Arrhenius rate equations 3 to 6.

$$dx/dt = A \times e^{\left(-E_a/RT\right)} \times (1 - x)^n \qquad (3)$$

where x = degree of conversion; t = time; dx/dt = reaction rate; n = reaction order;
A = pre-exponential factor; Ea = activation energy; R = gas constant; T = temperature (K)
The above expression in log form gives

$$\ln[1/(1 - x)^n] = \left(-E_a/R\right)\left(1/T\right) + \ln A$$
$$- \ln\left(dx/dt\right) \qquad (4)$$

For a degree of conversion, *xi,* under temperature, *Ti,*

$$\ln[1/(1-x_i)^n] = \left(-E_a/R\right)\left(1/T_i\right) + \ln A$$
$$- \ln\left(dx_i/dt\right) \qquad (5)$$

Since the reaction rate is constant,

$$\ln A - \ln\left(dx_i/dt\right) = \beta \text{ (constant or intercept)} \quad (6)$$

Therefore, a plot of the logarithm of the heating rate $\ln[1/(1-x_i)^n]$ versus reciprocal temperature $\left(1/T_i\right)$ gives a straight line with a slope equal to $-E_a/R$ and an intercept equal to β, assuming a first order reaction. TGA/DTA of materials may be determined under conditions that accelerate its rate and the resulting parameters extrapolate to predict a service lifetime for useful commercial importance.

3.4 Morphological Study Using Scanning Electron Micrograph

Surface morphology and analysis of microstructure characteristics of both pure and modified bitumen samples were assessed using Hitachi S-4700 field emission scanning electron microscope (FE-SEM). The Hitachi S-4700 FE-SEM produces cold field emission and high-resolution micrographic pictures. Bitumen specimens were flash frozen using liquid nitrogen at temperature of -26⁰C and at 30Pa pressure. Micrographic images were taken using cryogenic stage 15-kV electron beam at 2000x desired magnifications.

4. RESULT AND DISCUSSION
4.1 Short-term Loading (Uniaxial Static Creep Test)

Static creep evaluation was conducted to assess the short term deformation of asphalt to static loading and unloading with time under a particular temperature. The deformation responses of polymer asphalt mixtures (0, 1, 2 and 3% PP) are in Table 1. Figures 1 to 4 show the maximum creep deformation, creep recovery or rebound and permanent creep deformation for the various mixes.

The various trends emanating from this test are as follows:

- The strain is temperature dependent, that is, the higher the temperature the higher the strain. The creep strain consists of an instantaneous part and a time dependent part and the deformation is partially recovered. At high load levels or high surrounding temperatures, creep deformation becomes plastic and can increase several times than instantaneous deformation thereby leading to premature failure of structure [48].

- The presence of HDPP in the asphalt mixes from 0 to 3% minimized creep strains even at the higher test temperature because of temperature resistivity of the polymer. Creep strains in Figures 1 to 4 decreased as HDPP increased from 0 to 3% and for 10°C to 60°C temperature range considered. Higher molecular weight in HDPP and aromatic rings in bitumen add to thermal stability, thereby increasing the creep resistance of a polymer-bitumen mixes [49].

Table 1: Summary creep deformation test result for wet process

Temperature (°C)	HDPP Content (%)	Maximum Creep *10⁻³ (mm/mm)	Creep Recovery *10⁻³ (mm/mm)	Permanent Creep *10⁻³ (mm/mm)
10	0	1.8223	0.2416	1.5807
	1	1.6881	0.2958	1.3923
	2	1.5600	0.259	1.3010
	3	1.3995	0.2322	1.1673
25	0	4.5143	0.3323	4.1820
	1	4.1820	0.7188	3.4632
	2	3.8642	0.6070	3.2572
	3	3.4669	0.5000	2.9669
40	0	9.7913	2.0477	7.7436
	1	8.7218	1.8241	6.8977
	2	8.0590	1.4944	6.5646
	3	7.2305	1.5532	5.6773
60	0	11.6543	1.7322	9.9221
	1	10.2730	1.7257	8.5473
	2	9.2371	1.0704	8.1667
	3	8.2875	1.0950	7.1925

- It was also observed that shear deformation due to viscous behaviour that renders asphalt mix susceptible to rutting is lowered at both lower and higher temperature with increasing polymer content and the finding is consistent with [50-52].

4.2   Long-term Loading (Uniaxial Static Creep Test)

The results of long term loading are shown in Figure 5. The following deductions from long term loading are made:

- The deformation induced strains in the first phases are instantaneous and could be associated with volume change, and compaction of asphalt concrete. This view is supported by [53].   The deformation only ended at secondary phase showing constant slow rate of increase in rutting with increase in shear stressed and did not progress to tertiary phase as polymer content increases from 0 to 3% HDPP and for the duration of testing. Researchers have observed that tertiary stage exhibits high level of rutting and is related to plastic deformation with flow under no volume change [2; 54-55].
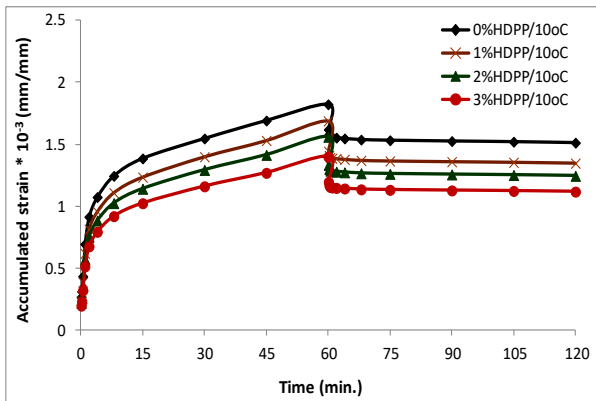
Figure 1: Relationship between accumulated static creep with time for specimens at 10°C
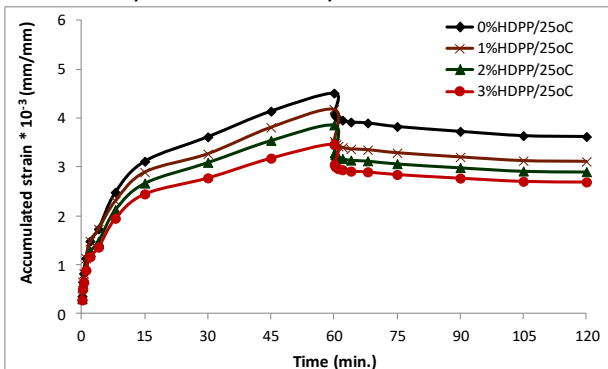

Figure 2: Relationship between accumulated static creep with time for specimens at 25°C
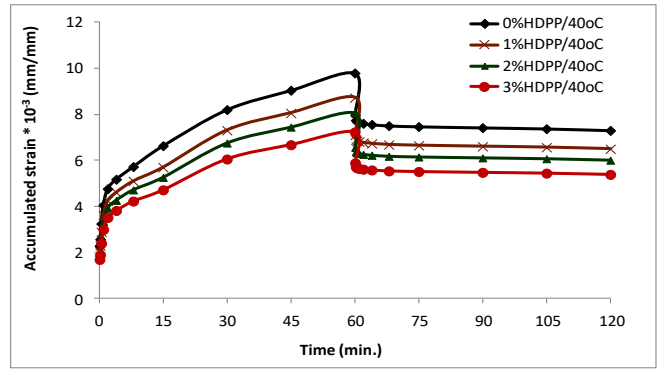

Figure 3: Relationship between accumulated static creep with time for specimens at 40°C
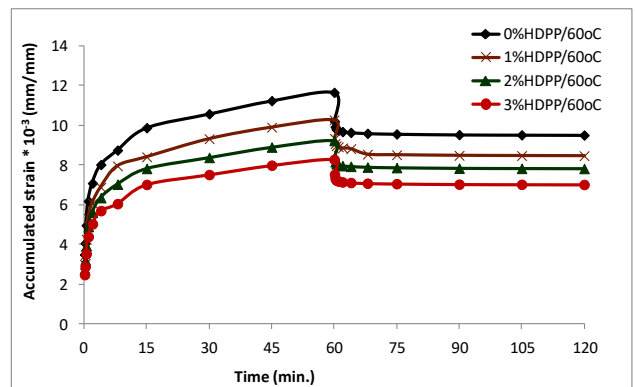

Figure 4: Relationship between accumulated static creep with time for specimens at 60°C
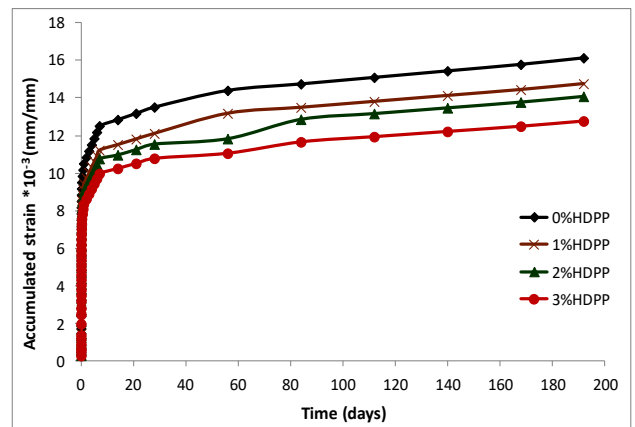

Figure 5: Long time accumulated static creep for specimens at 25°C

4.3 Thermal Gravimetric Analysis

TGA and DTA results in Figures 6 and 7 respectively show the trends of degradation and phase transition of unmodified and HDPP modified bitumen. The deductions made from the results are:

- The result in Figure 11 indicates that at 450°C, for instance, the TGA weight losses of 42.2%, 29.6%, 27.9% and 24.5% respectively for 0, 1, 2 and 3% HDPP contents. ASTM D4124-09 separated bitumen

constituents according to molar mass, solubility and polarity of fractionates called SARA (S-Saturates, A-Aromatics, R-Resins, and A-Asphaltenes) [56]. The first three together forms light molecular weight Maltene component and mostly leads to ageing as they volatized, but decomposition or oxidation of heavy molecular weight asphaltene component further worsen ageing and deformation [57].

- According to [58], polymer-bitumen mix forms chemical bonds between the asphaltene compounds and strong bonding of the diene molecules from the polymer and increases thermal stability. The results agree with the finding by [59] that the main combustion and phase transition of bitumen lies in the exothermic reaction second phase at temperature ranging from 405 to 490°C where the main weight loss occurred.

- The trends supports that HDPP bitumen has lower weight loss, lower rate of degradation and volatization and more temperature resilience; thus, could improve the rheology and longer lifespan better than pure bitumen [60].

- Although, EN12591 recommends that the maximum temperature of bitumen at any stage of mix preparation to be 180 °C for 50/70 penetration grade, active degradation occurs between temperature of 250°C to 550°C where components such as saturates and aromatics are volatized and asphaltene decomposed [61]; [16]; [62]. Addition of HDPP leads to increase in the solid than fluid components [63]. This induces more temperature resistivity as the ability to lose light end components decreased [43] and thus, increasing the melting temperature of the blend containing HDPP polymer [64].

4.4 Morphological test using Scanning Electron Micrograph (SEM)

The micrograph images of 0% and 2% HDPP bitumen samples are shown in Figure (8a) and (8b). Figures 9a and 9b shows the fibre histogram while Figures 10a and 10b are the pore histograms of the two samples respectively. The following observations were made from the results:

- The micrographs were taken at same resolutions and magnifications. Plate 1(0% HDPP) has larger pore areas ranging from 0.41- 1668.91μm^2 (Figure 15) whereas Plate 2 (2% HDPP) has a pore range

of 0.1-182.88 μm^2 (Figure 16). The smaller the pores, the stronger the bond and strength of the material. According to [65], a compatible mixture of polymer and asphalt gives better morphological and thermal properties than unmixed asphalt. The morphological study showed that 2% HDPP bitumen imparts more on the overall strength and stability of the asphalt mix than the control (0% HDPP).

- Also, the histogram of fibre length showed the 0% HDPP bitumen is between 2.13 to 18.85μm (Figure 13) while 2% HDPP bitumen is ranging from 834.19nm to 7.78μm (Figure 14). It showed that 2% HDPP bitumen has more reaction surface due to surface area than the control (0% HDPP bitumen). Elasticity and strength of unmodified bitumen may be sufficient to resist the stresses that traffic places on the pavement. The dynamic interaction between polymer dispersed bitumen matrix coalesce the structure and reinforces the strength [66, 67].
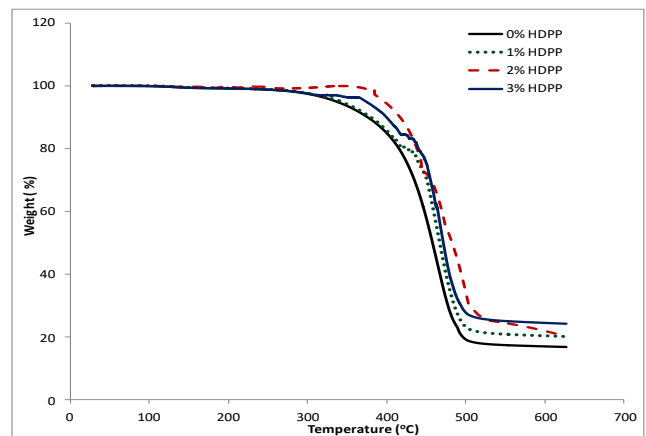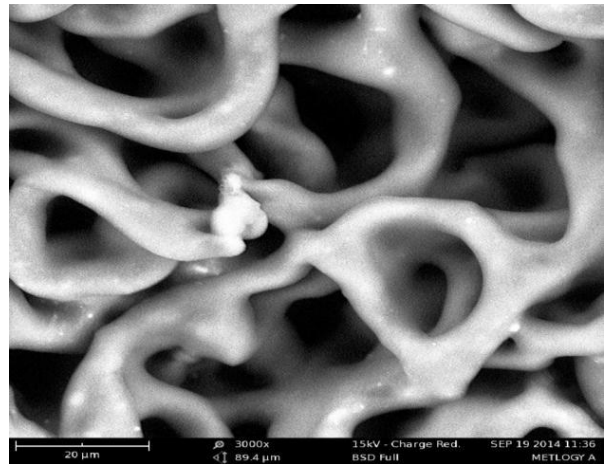

Figures 6: TGA result of HDPP polymer bitumen
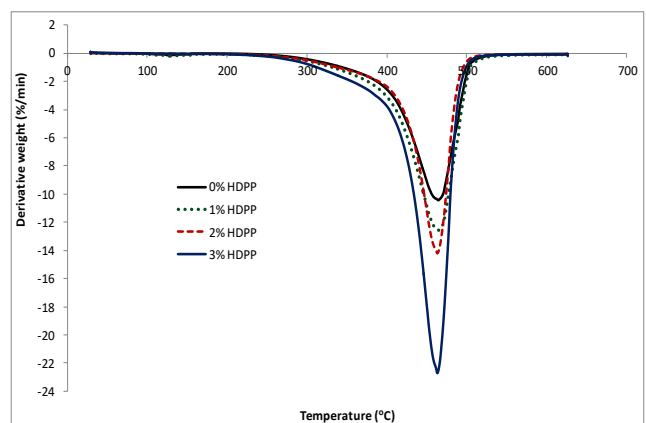
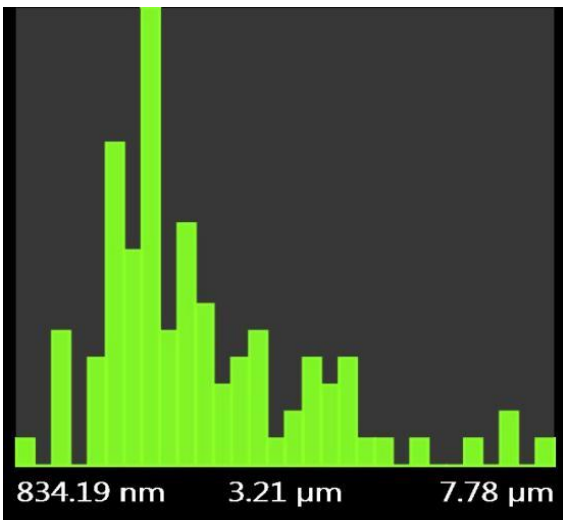*Figures 7: DTA plots of HDPP polymer bitumen*
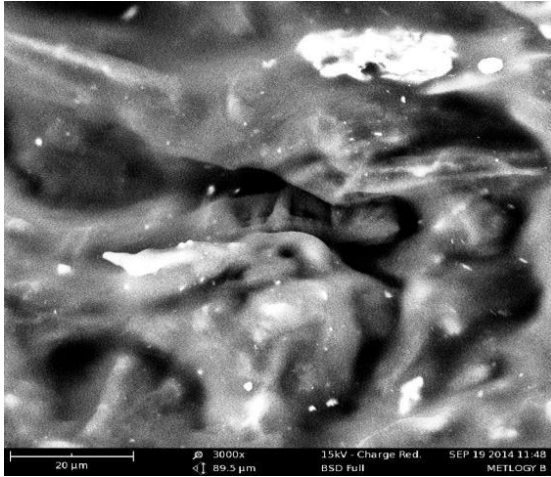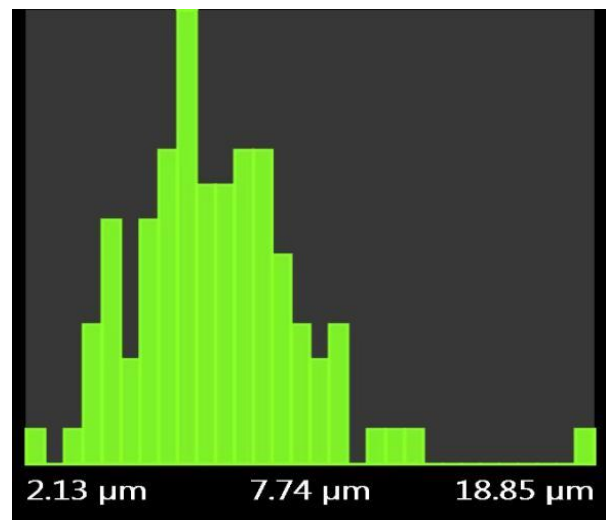
(a)   (b)

Figures 8: a) Micrograph of 0% HDPP bitumen b) Micrograph of 2% HDPP bitumen
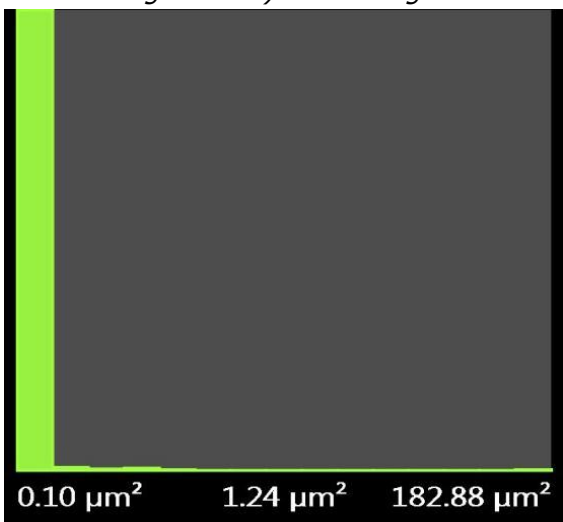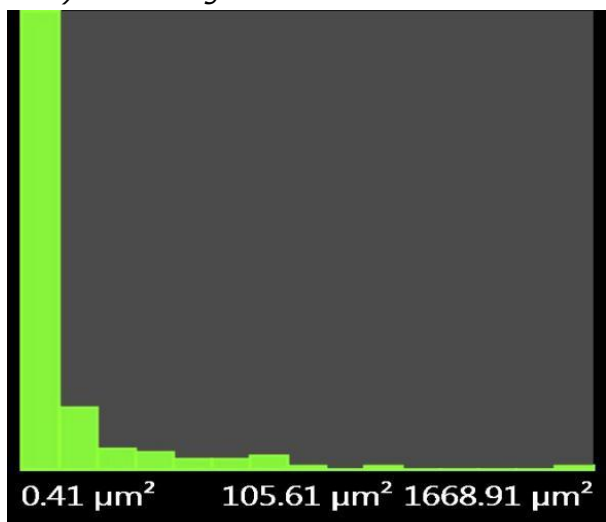

(a)   (b)

Figures 9: a) Fibre histogram of 0% PP bitumen b) Fibre histogram of 2% PP bitumen

(a)   (b)

Figures 10: a) Pore histogram of 0% PP bitumen b) Pore histogram of 2% PP bitumen

5. CONCLUSION

From the outcome of the study, the following conclusions are hereby made:

- For the short term loading wet process, the lowest creep strain at maximum stress being optimum creep resistance lies at 3.0% HDPP whose value is $1.3995*10^{-3}$ (mm/mm) for low temperature of 10°C and $8.2875*10^{-3}$ (mm/mm) for 60°C field temperature. The maximum creep strains for 0% HDPP (control) are $1.8223*10^{-3}$ (mm/mm) and $11.6543*10^{-3}$ (mm/mm) respectively for 10°C and 60°C. These values account for 23.2% strain reduction at 10°C and 28.9% strain reduction at 60°C and as such impart better creep resistivity for HDPP asphalt wet mix than the control.

- Permanent creep strains decreases as HDPP content increased from 0-3%, but generally, there are increasing strain trend with increasing temperature from 10°C to 60 °C since flow is increased by higher temperature. The result of long term loading for the period of 192days shows that accumulated creep strain for 0% HDPP (control) is $16.1348*10^{-3}$ (mm/mm) while 3% HDPP accumulated $12.7715*10^{-3}$ (mm/mm) accounting for 20.9% creep strain reduction.

- At the critical degradation temperature range of 250°C to 550°C, 2% HDPP modified bitumen has better resilience than 0, 1 and 3% HDPP. The shape and dispersed structure of 2% HDPP modified bitumen has better morphology than the control. Polymer modified bitumen produce better morphological, temperature resistivity and creep deformation resistivity at optimum HDPP content of 2.0% and has rheological and mechanical properties to increase pavement longevity.

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