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Value-Added Application of Waste PET Based Additives in Bituminous Mixtures Containing High Percentage of Reclaimed Asphalt Pavement (RAP)

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Abstract

The use of reclaimed asphalt pavement (RAP) in road pavement construction has been widely encouraged due to its environmental and economic benefits. However, the percentage of RAP is usually capped at low percentages as studies have shown that a high percentage of RAP might be detrimental to overall pavement performance. Recent research has shown that the addition of waste plastic materials such as Polyethylene Terephthalate (PET) or their functionalized additives into asphalt pavement may potentially improve the durability of pavement and also help alleviate the environmental problems caused by plastic. The main objective of this study is to investigate the feasibility of using the additives, derived from waste PET through an aminolysis process, to improve the performance of bituminous mixtures containing RAP, by characterising the binder properties. To achieve this objective, binder samples composed of virgin bitumen, aged bitumen at various percentages, and PET derived additives, were prepared. These samples were then characterized through various laboratory tests, including dynamic shear rheometer, bending beam rheometer, moisture susceptibility, infrared red spectroscopy and fluorescence microscopy tests. The results indicated that the samples containing RAP and PET derived additives provided better overall performance compared to the conventional binder, increasing the rutting resistance by at least 15% and fatigue cracking resistance by up to 60%. Usage of such waste PET based

additives as an additive for RAP mixtures represents an approach to deal with a relevant recycling problem while simultaneously recovering two value-added materials.

Keywords: Waste PET; RAP; Chemical Recycling; Sustainability

1. Introduction

The inclusion of waste materials such as reclaimed asphalt pavement (RAP) in pavement mixtures has become increasingly common due to numerous environmental and economic benefits. Practitioners around the world are constantly assessing the advantages of allowing higher percentages of RAP in pavement while also maintaining the highest performance standards to meet the increasing demands and regulations (Al-Qadi et al., 2007). Many transport authorities and departments have limited the maximum amount of RAP used in surface layers, certain mixtures types and in some cases large or critical projects. The amount of RAP used in surface layers was usually less than 15 percent initially as there was no significant advantage economically for using a larger percentage of RAP. In 2006 and also in 2008, there was a sharp increase in asphalt binder costs and reduction of supplies, hence using a greater percentage of RAP became a priority again (NCDOT, 2007). RAP has usually undergone years of natural ageing and the binder that is present in RAP is aged and harder, hence the incorporation of this aged binder to virgin HMA material results in a modified mix that is potentially also harder (McDaniel and Shah, 2003). While this may enhance certain performance properties such as rutting resistance, it also raises significant concerns over fatigue cracking and moisture damage performance (MTO, 2008). Additionally, there are also concerns over RAP mixture design especially in regard to the amount of RAP binder that is mobilised in a mixing plant or in the mix design process. Although it is regarded that complete mobilisation and blending is unlikely for traditional asphalt mixtures, it is recognized now that a considerable amount of mixing occurs. RAP mixtures behave as a composite of both the RAP and virgin binders (Sreeram et al., 2018). Due to these limitations, it now common that RAP mixtures are used in conjunction with other bitumen modifiers such as rejuvenators and antistripping additives to improve its rheological properties and provide a more balanced performance. Many of these commonly used modifiers are polymeric in nature and its incorporation in RAP mixtures are known to enhance its rheological properties (Hagos et al., 2012). Specifically, this study is concerned with the development of polymeric additives through the chemical recycling of waste PET to improve the performance of mixtures containing RAP, by characterising the binder properties.

2. Polymer Modification in RAP mixtures

Currently, there is an increase in the usage of polymer modified bitumen modified bitumen in road pavement and highways as they offer considerable advantages over conventional bitumen such as lower susceptibility to temperature variations and higher resistance to deformation at elevated pavement temperatures (Yildrim, 2007). The addition of these polymers such as styrene–butadiene–styrene (SBS), ethyl vinyl acetate, polyvinyl chloride, polyethylene, polyoctenamer, polyethylene terephthalate(PET) materials in asphalt pavement has now been widely studied and its benefits extensively established. The use of polymers in asphalt binders generally improves the deformation performance of HMA mixes (Kim et al., 2014), however when polymers are used in conjunction with RAP, the behaviour of the resulting mix can vary considerably depending on the type of polymer that is used. Published literature has indicated mixed results when considering the use of RAP with polymers. It has been indicated that at lower levels of polymer modification the addition of RAP results in increased rutting resistance while at higher levels of polymer modification the addition of RAP does not have a significant effect on the rutting resistance of the HMA mix (Kodippily et al., 2017). The main limitation of using RAP in HMA is the low temperature cracking performance due to additional stiffness of the combined mixture. Polymer modification has been extensively used to improve the cracking performance of HMA mixes where the presence of polymers enhances the elastic properties of asphalt binder thus improving the bond strength between the materials and prolonging the service life of the pavement. Overall, polymer modification has been shown to reduce the low temperature cracking susceptibility of HMA mixes while enhancing the fatigue performance, hence making it suitable to be incorporated into RAP mixes to improve its performance properties (Mogawer et al., 2011). Polyethylene Terephthalate (PET), a thermoplastic polyester which constitutes 18% of the total polymer produced worldwide (Ji, 2013) and commonly used to make plastic bottles is used as the polymeric additive in this research. The large-scale use of PET for manufacturing of plastic material in various forms including bottles has made it a major cause for environmental pollution. This material has also been known to transport persistent organic pollutants such as polychlorinated biphenyl (PCB) which can have negative effects on the environment. The slow rate of decomposition of PET and its non-bio degradable nature has led to scientists looking into ways to recycle it. Currently, both chemical and physical methods have been employed to recycle PET however chemical recycling has the advantage of leaving the polymer to produce several industrially useful products and eliminating the practice of disposal of PET in landfills. Since PET recycling has not been carried out in the same amount as its production; it would be worthwhile to find out new application areas for PET bottle wastes to maximize their end-of service life management effectiveness (Sarker and Rashid, 2013). The usage of waste PET as an additive for asphalt road pavement material has been studied previously although it can still be regarded to be at an early stage. In prior studies, PET waste was generally added to the asphalt mixture with dry process or used as aggregate in the asphalt mixture in order to improve the resistance to permanent deformation, Marshall Stability, stiffness and fatigue life of road pavement (Ahmadinia et al., 2011; Moghaddam et al., 2014). However, phase separation and a decrease in specific gravity of the asphalt mixtures have been reported due to the inhomogeneous distribution of PET in asphalt (Ameri and Nasr, 2016). Apart from this, asphalt was modified with a number of additives derived from PET waste by aminolysis and glycolysis reaction as a wet process and found to improve the Marshall Stability and moisture resistance depending on the asphalt and additive contents (Padhan et al., 2013; Guru et al., 2014). These initial studies have indicated that the PET additives have significant potential to improve the stripping characteristics and overall performances of asphalt mixtures. It is likely that PET additives could act similar to commercial rejuvenating agents and hence would be suitable to be incorporated into high RAP mixes to improve moisture damage, fatigue cracking and low temperature performance properties. The combined use of waste plastic and reclaimed asphalt pavement could also open a new chapter in the efficient disposal of products currently landfilled and help alleviate the pressure of disposal. In this study, Triethylenetetramine (TETA) was utilised for the aminolysis of waste PET into an additive and used for bitumen modification. Subsequently, the modified binders were tested for various performance parameters to evaluate its rheological properties.

3. Material Preparation and Research Methodology

3.1. Material Preparation

3.1.1 Poly (ethylene terephthalate) (PET)

The waste PET water bottles were collected after proper identification. The PET bottles were stripped of all labels and cleaned with normal detergent solution followed by proper washing and drying to remove any potential contaminants. The bottles were then cut into small pieces about 5mm by 5 mm and dried at 80°C temperature for 4 hours.

3.1.2 Extraction of Aged Binder from Reclaimed Asphalt Pavement (RAP)

The reclaimed asphalt pavement (RAP) was obtained from the Highways Department, Hong Kong. It was obtained by 10 mm wearing course cold milling and was reported to be of low to moderate ageing level. The RAP binder was extracted using the solvent extraction method as per AASHTO T164.

3.1.3 Synthesis of PET Additive

The PET waste was used as a synthon and chemically converted through a non-catalytic route into benzamide derivatives through an aminolysis process (Leng et al., 2018). A three necked 500 ml round bottomed flask equipped with a heating mantle, overhead stirrer, water condenser, nitrogen gas sparging tube and a thermos well pocket containing a thermometer was charged with 30g of PET and excess triethylenetetramine (TETA) in the presence of nitrogen gas. The mixture was heated at 130°C to 140°C to reflux for 2 hours. The solution turned homogeneous as the PET degradation completed. At the end of the reaction, the polyamines and glycols are recovered under vacuum. The resulting product is a residue recovered in quantitative yields at ambient temperatures (Figure 1&2).

Figure 1. Scrap PET

Figure 2. PET Derived Additive

3.2. Characterization of PET additives

The PET degradation was confirmed by IR analysis which showed the disappearance of ester group peak at 1735 cm^{-1} and the formation of amide peaks at 1637.6 cm^{-1} and 1544.4 cm^{-1} , confirming the substitution of amines by TETA amide (Figure 3).

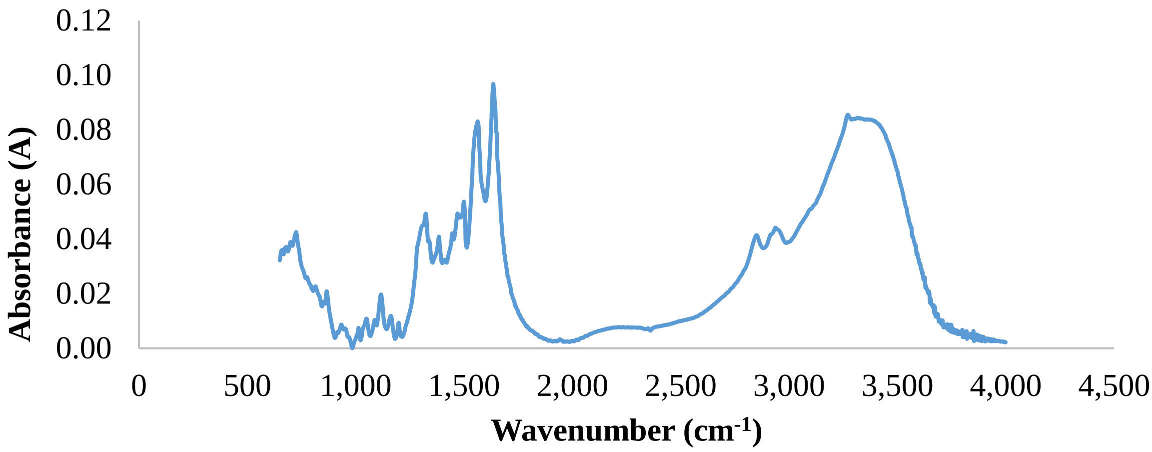


Figure 3. FTIR Spectrum of PET Additives

The virgin PET and synthesized PET additive was also studied under a scanning electron microscope to ascertain its morphology and change in microstructure after synthesis. It can be observed that the PET surface has undergone complete degradation after the aminolysis reaction (Figures 4&5). This degradation is important in terms of performance and also effective mixing in asphalt.

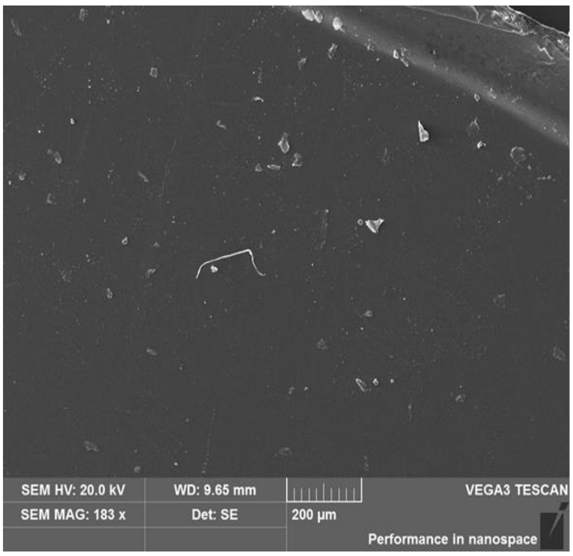


Figure 4. SEM Image of Scrap PET

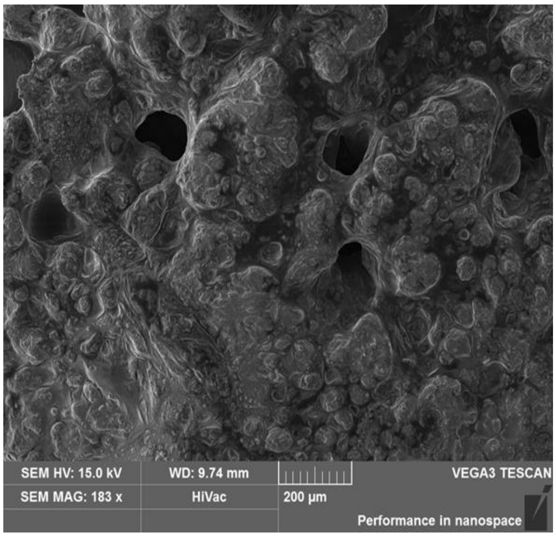


Figure 5. SEM Image of PET additives

3.3. Mechanism of Synthesis

PET possesses a labile ester group in every repeating unit. These ester groups are prone to solvolysis reaction by active polar species such as the free amines present in the triethylenetetramine (TETA). As represented by the chemical reaction in Figure 6, the amine group of TETA reacts with the ester group of PET to form the PET derived additive.

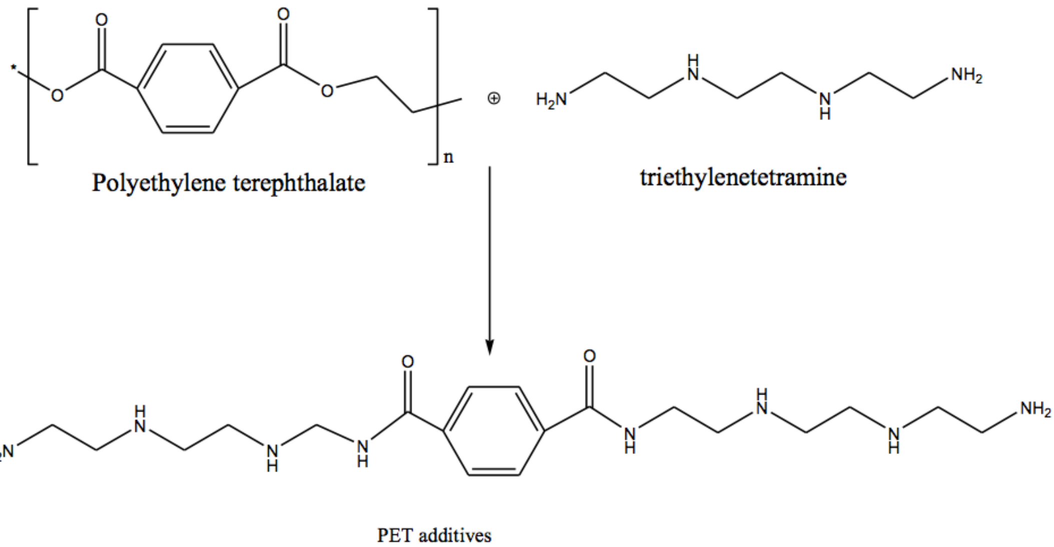


Figure 6. Typical Synthesis of PET Additive through an Aminolysis Process.

3.4. Preparation of Modified RAP and PET asphalt binder

The optimum percentage of PET additives used for this study was chosen based on prior literature and extensive antistripping tests (Padhan et al., 2013). Two control samples of virgin binder, virgin binder with 2% PET additive (PET modified binder) and PET modified binder mixed with 15%, 25% and 40% RAP binders were prepared using a high shear mixer at around 150^{o}C for 2 hours at a mixing rate of 4000 r/min. Table 1 shows a sample criterion for the selection of the optimum percentage of PET additive used for this research study. The percentage (%) of antistripping characteristics in Table 1 refers to the amount of binder that has not been stripped from the granite-based aggregate after hot water immersion tests in accordance to ASTM -D3625 standards.

Table 1. Selection Criteria for % of PET Additive Used Based on Hot Water Stripping Tests

Binder Composition	**Percentage (%) of Antistripping**
100% RAP binder	40 to 45
25% RAP binder + PET (1%) modified binder	65 to 70
25% RAP binder + PET (1.5%) modified binder	75 to 80
25% RAP binder + PET (2%) modified binder	85 to 90
15% RAP binder + PET (2%) modified binder	90 to 95
40% RAP binder + PET (2%) modified binder	75 to 80

4. Experimental Methods

The flowchart below (Figure 7) shows the experimental program of this study. To better understand the working mechanism of the PET additive in the asphalt binder, chemical characterisation tests such as Fourier Transform infra-red (FTIR) and Fluorescence microscopy (FM) tests were also conducted in addition to conventional binder tests.

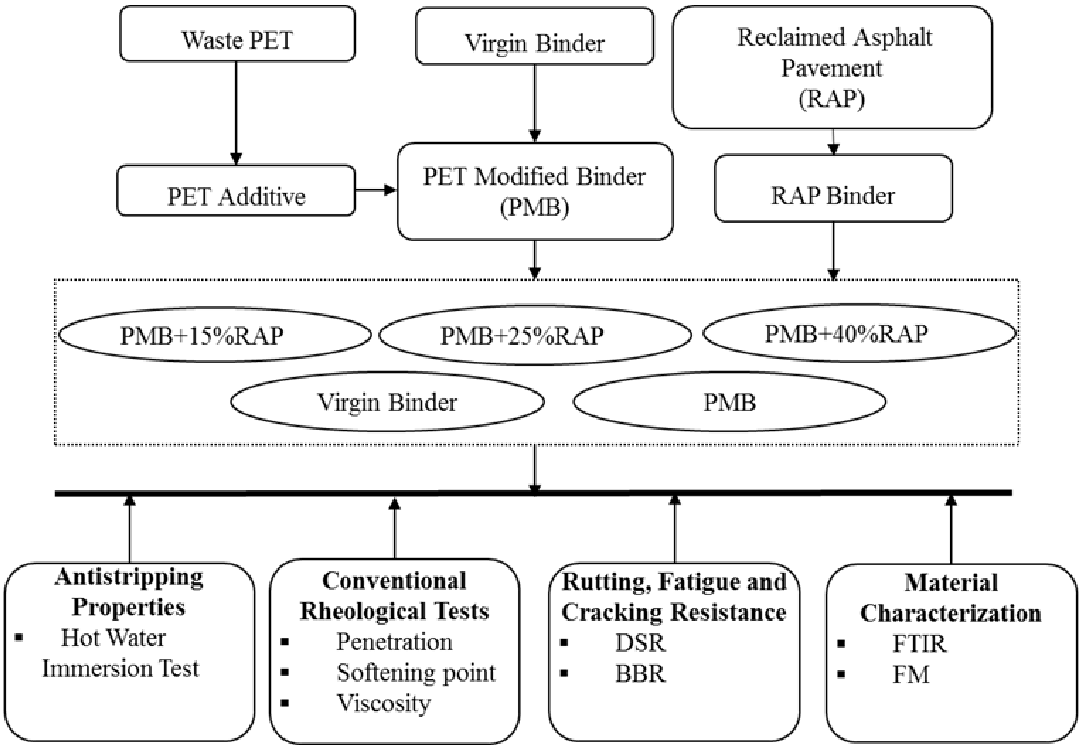


Figure 7. Flow Chart of the Experimental Program

4.1. Penetration, Softening Point and Ductility Tests

The intermediate test temperature properties, including penetration at 25°C, softening point and ductility at 25°C, were tested in accordance with ASTM methods D5, D36 and D113 (0072) respectively.

4.2. Brookfield Viscosity Test

To obtain the viscosities of various binders, Brookfield viscosity tests were conducted using a DV-II Brookfield rotational viscometer, in accordance with AASHTO T316. All the asphalt test samples were conditioned in the thermo-container for about 30 min at the desired test temperature before carrying out the test. The viscosities of the various binders were measured at 3 different temperatures including the mixing temperature.

4.3. Dynamic Shear Rheometer (DSR)

The DSR PG grading tests were conducted at a fixed frequency of 10 rad/s and at various temperatures ranging from 58°C to 76°C using an MCR 702 dynamic shear rheometer, manufactured by the Anton Paar Inc. The gap distance between the two circular plates of the

DSR was set at 1 mm by employing a 25mm plate diameter for unaged and RTFO aged binder samples while pressurised ageing vessel (PAV) samples were used for fatigue evaluation with 8mm plate diameter and 2mm gap. The frequency sweep tests were conducted on the samples using 25mm diameter plates and 1 mm gap with a frequency ranging from 0.1 to 100 Hz at 60°C.

4.4. Bending Beam Rheometer (BBR)

In order to assess the impact of waste PET derived additives on the low temperature performance of RAP modified asphalt, BBR tests were conducted at -6°C, −12°C and −18°C using a Cannon TE-BBR instrument.

4.5. Fourier Transform Infrared Spectroscopy (FT-IR)

To acquire the infrared spectra of PET modified binders, the FTIR test was conducted using an IS50 FTIR manufactured by the Thermo Fisher Scientific Inc. Asphalt samples were prepared by covering a thin asphalt film on the surface of ATR crystal. The wavelength range chosen for each test was from 600 cm^{-1} to 4000 cm^{-1}.

4.6. Fluorescence Microscopy (FM)

Fluorescence microscope (FM) is a microscopic method that enables the observation of polymer distribution in asphalt binder. The FM test was conducted for the various samples using a Leica TCS SPE confocal microscope. The sample preparation involved placing a thin uniform layer of binder on a glass slide covered by a cover slip.

5. Results and Discussion

Table 2 illustrates the conventional binder properties of the various samples. The penetration test at 25°C, the softening point tests and the ductility tests were conducted for the RAP binder, virgin binder and the PET modified binders. The PET modified binder was characterised by a small increase in softening point and decrease in penetration value as compared to the virgin binder. The addition of the harder RAP binder further heightened this trend. (Figure 8)

Table 2. Conventional Binder Properties

Properties	Virgin Binder	PET Modified Binder	PET Modified Binder + 15% RAP	PET Modified Binder + 25% RAP	PET Modified Binder + 40% RAP	RAP Binder
Penetration (0.1 mm)	69	54	51	46	44	39
Softening Point (°C)	48	49	50.5	51	52.5	57.5
Ductility (cm)	100+	100+	100+	100+	100+	85

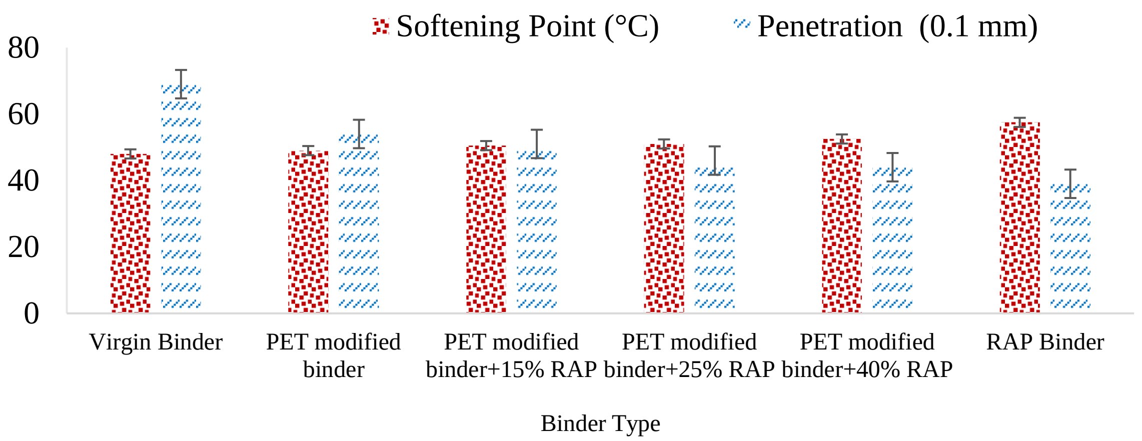


Figure 8. Softening Point and Penetration Values

5.1. Viscosity

The viscosity test was carried out using at various temperatures to ascertain the change in viscosity of the binders. As expected, the aged RAP binder has a much higher viscosity than the virgin binder. This is predictable as the asphalt binder in RAP loses light components during service. The viscosities of all PET modified asphalt binders decreased with the increasing test temperature and increased with increasing RAP content. The PET modified binders have comparable viscosity to the virgin binder at the mixing temperature of around 150°C (Figure

9). The inclusion of the moderately aged RAP does not significantly hinder the workability of the modified binders.

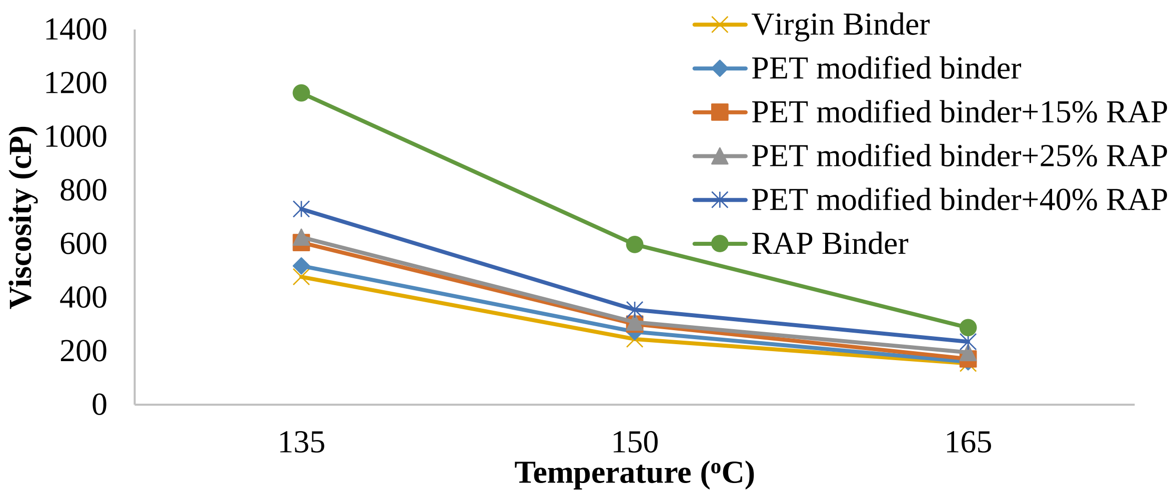
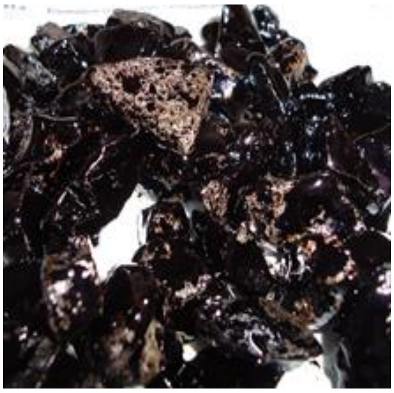


Figure 9. Brookfield Viscosity of Virgin and Modified Asphalt Binder

5.2. Antistripping Properties

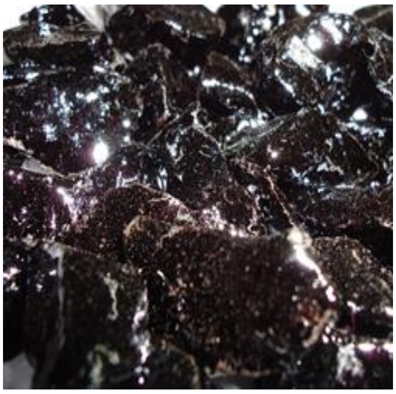
The most commonly used tests to ascertain the antistripping properties include the hot water immersion test and the Marshall stability test. The samples were tested for the hot water immersion tests as per ASTM D3625M-12 using locally available granite-based aggregates and some of the results are shown in Figure 10. The PET modified virgin binder is the most resistant to stripping owing to the addition of the antistripping additives. The addition of 15% and 25% RAP binder did not deteriorate the stripping characteristic of the PET modified binder to a significant extent and was even comparatively better than the virgin binder. The addition of the PET additives having a terminal amine group facilitates the improvement in stripping performance of the oxidised binder. This amine group assists the binder and aggregates to bond significantly better thereby increasing its antistripping characteristics.

a) RAP Binder

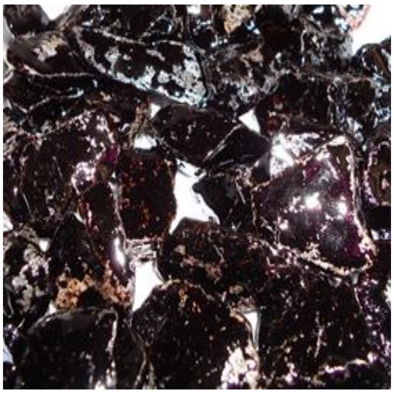


b) Virgin Binder

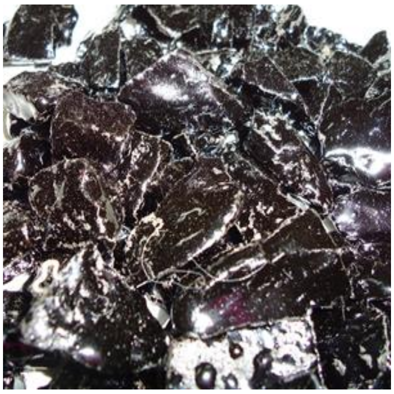
c) PET Modified Binder



d) PET Modified Binder with 15% RAP



e) PET Modified Binder with 25% RAP



f) PET Modified Binder with 40% RAP

Figure 10. Photos Taken after Hot Water Immersion Testing

5.3. Dynamic Shear Rheometer (DSR) Test

The DSR test was conducted to evaluate the permanent deformation and fatigue cracking properties of the different mixtures. Physical property measurements were made for the original binder samples, rolling thin film oven (RTFO-aged) samples and the pressurized ageing vessel samples (PAV). According to the Superpave specifications AASHTO T316, the minimum value of G*/Sinδ (rutting factor) should be greater than 1.00 kPa and 2.20 kPa for unaged and RTFO-aged binder samples respectively. In all DSR tests, the PET modified binders showed higher G*/sinδ values as compared to the virgin binder (Figures 11&12). When the stiffer RAP binder was added to the PET modified binder, there is a noticeable increase in G*/sinδ at all temperatures, i.e. 58 °C, 64°C ,70°C, 76°C. This indicates that RAP modified PET binders are less susceptible to rutting or permanent deformation at high temperatures as compared to the virgin binder. When considering the DSR results for the PAV aged samples (Figure 13), G*Sinδ is the fatigue resistance parameter which according to the Superpave asphalt binder specification, should have a maximum value of 5000 kPa. Lower values of G*Sin(δ) are desirable from the standpoint of resistance to fatigue cracking. The G*sinδ obtained in this study were lower for the PET binder modified with RAP signifying better performance to fatigue cracking as compared to the virgin binder.

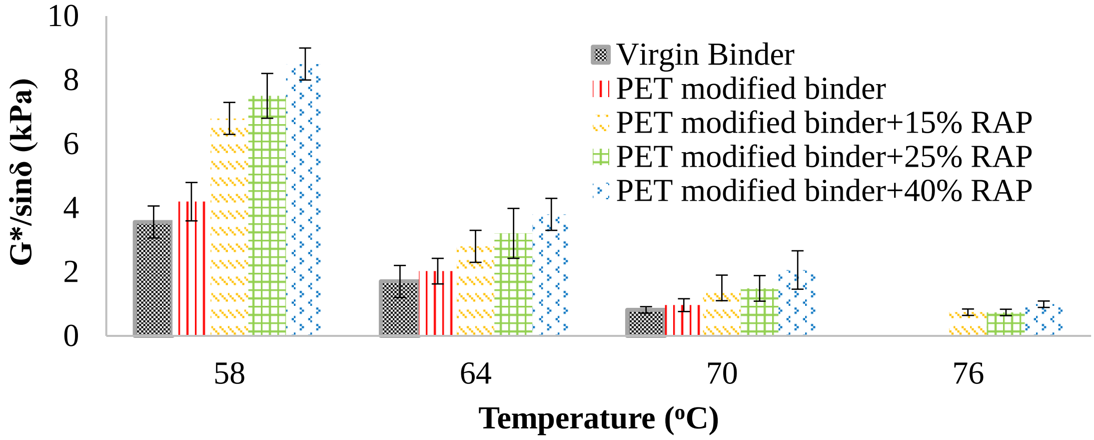


Figure 11. G*/sinδ vs Temperature for Original Binder Samples

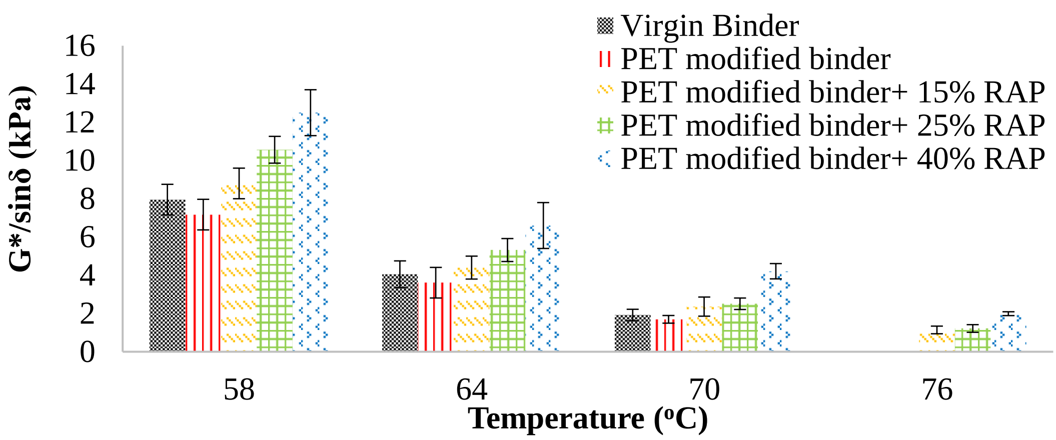


Figure 12. G*/sinδ vs Temperature for RTFO-aged Samples

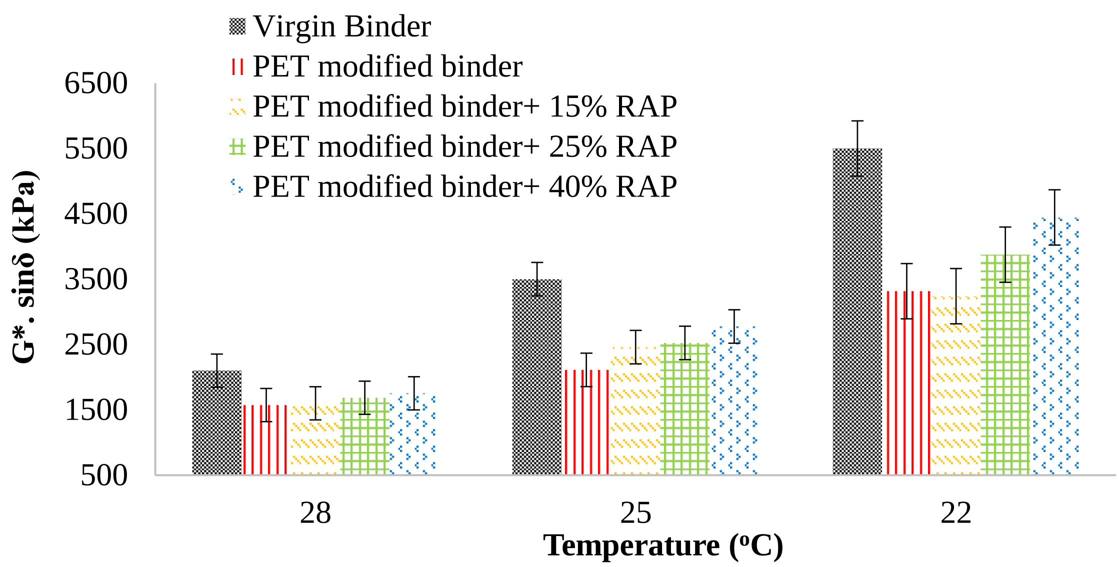


Figure 13. G*sinδ vs Temperature for PAV Samples

The PET additive had a substantial effect in improving the fatigue cracking properties of RAP mixtures and behaved in a similar manner to commercial rejuvenating agents. The chemical properties of such PET amides can be varied by changing the number of amine groups and their positions in the molecule. In addition, the balance between the length of the hydrocarbon chain and the number of amine groups has an influence on the adhesion strength and its solubility in asphalt. In this case, the presence of multiple amine groups may lead to the formation of complex molecular structures as shown in Figure 14. An increase in the length of the chain would also help to increase the tensile strength and elastic response, thus increasing fatigue performance. Many other researchers have also reported similar effects of amine based additives in the intermediate temperature zone, which allows the binder to dissipate energy without undergoing severe cracking (Kataware and Singh, 2017).

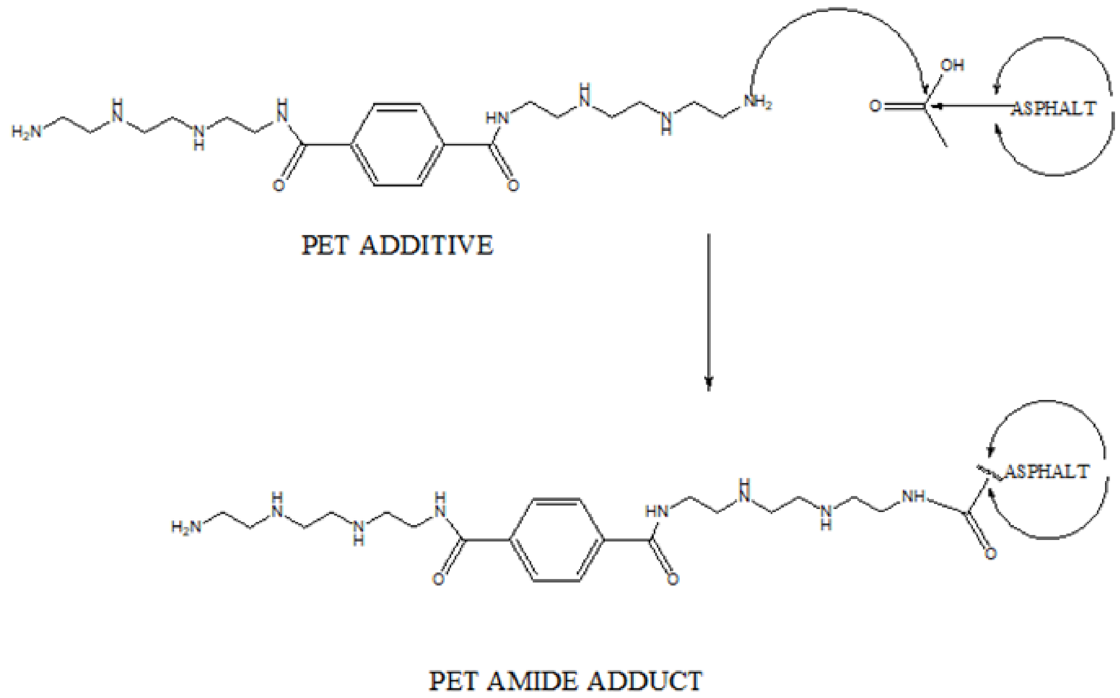


Figure 14. Interaction of PET Additives with Asphalt.

5.4. Frequency sweep test

The frequency sweep tests are generally conducted at various frequencies to identify the linear viscoelastic response of asphalt binders. The frequency sweep tests in this study was conducted on the various binders at a frequency ranging from 0.1 to 100 Hz at a temperature of 60°C. The tests indicated that all five binders have similar viscoelastic properties. With the increasing frequency, the complex modulus increased while the phase angle decreased. Compared to the virgin binder, all the PET modified binders were characterized with a trend of a noticeable increase in complex modulus and a slight decrease in phase angle which suggests that the incorporation of the PET additive leads to an enhancement in elastic property of the binder. This trend was more prominent with the addition of RAP in the PET modified binders. It can be thus inferred that the RAP binder used does not negatively affect the binders elastic character, this may be attributed to the textural compatibility and swelling potential of the PET additive in asphalt as discussed in the fluorescence microscopy section. (Figure 15&16)

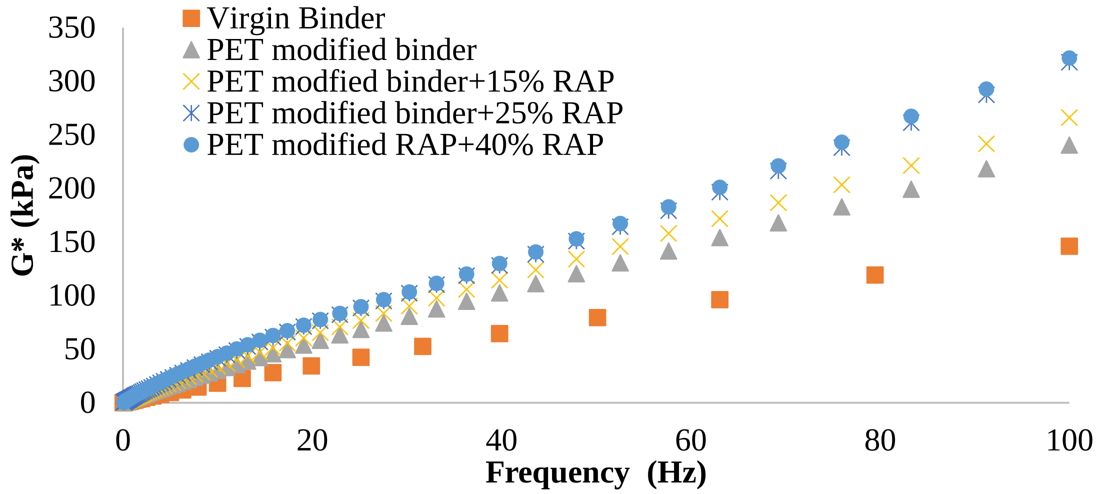


Figure 15. G* vs Frequency at 60°C

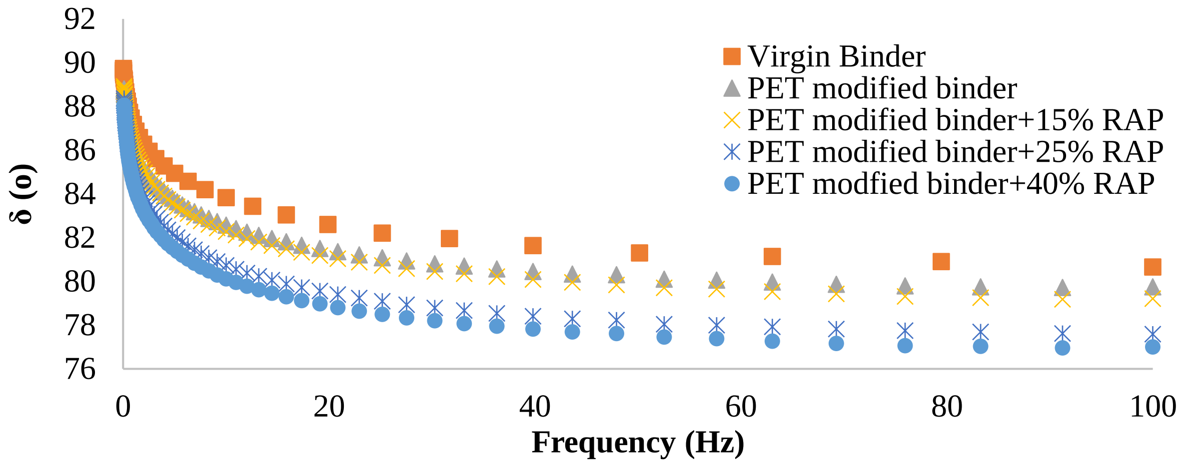


Figure 16. Phase angle (δ) vs Frequency at 60°C

5.5. Bending Beam Rheometer (BBR) Test

The BBR test was carried out at the required low temperatures of -6°C to -18°C for testing the low temperature cracking properties of the modified binders. This test is of significance as one of the main limitation of using RAP in HMA is its low temperature cracking performance due to the additional stiffness of the combined mixtures. Two parameters were obtained through this test; stiffness and the rate of change of stiffness with time (m-value). It was noted that all the binder samples performed similarly and met the creep stiffness specifications of less than 300 MPa up to -12°C (Figure 18). Similar trends are also observed for the m-values (Figure

17). A high m-value (> 0.3) is generally desired, as the temperature changes and thermal stresses accumulate, the stiffness will change quickly. A quick change in stiffness will cause the binder to shed stresses which can build up to a level where low temperature cracking could occur. Overall, the test results indicate that the addition of the PET additive improves the low temperature properties of the mixtures containing RAP components.

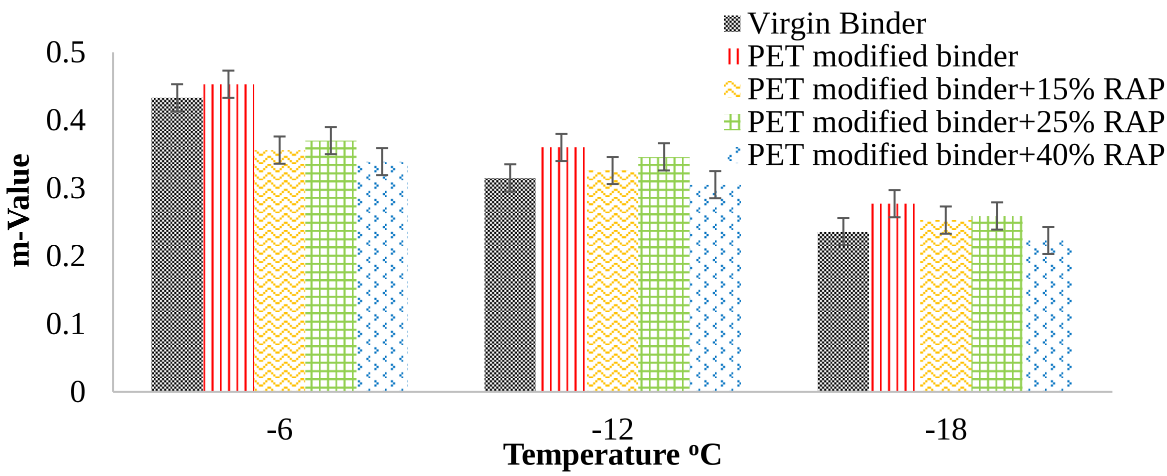


Figure 17. m-value Results vs Temperature

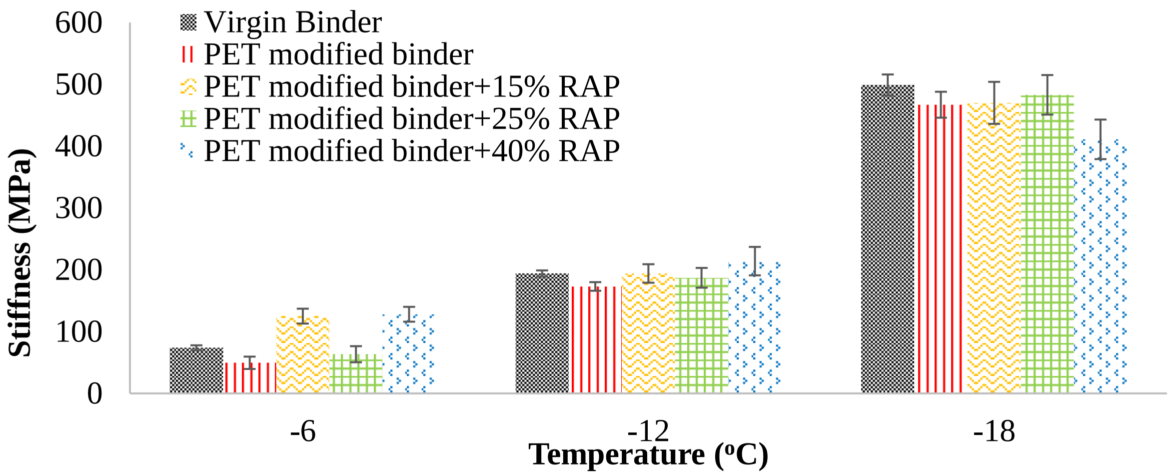


Figure 18. Stiffness vs Temperature

5.6. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The FTIR analysis was conducted on the samples to characterize the various functional groups in the original and modified binder samples. The aged RAP binder displayed physical and chemical changes as it was subjected to a thermal oxidative process over time, represented by the various increase in oxidative functional groups (Figure 19). This can be ascribed to the loss of volatiles or specimens of low molecular weight, or even the formation of hydrogen bonds. The formation of sulphoxide groups characterized by the band at 1030 cm-1 frequency (S=O stretching) was also noted. The absorption at the frequency 1160 cm-1 can be attributed to the anhydride groups formed after oxidation. Carbonyl groups were also observed at a frequency of 1700 cm^{-1} in the case of the RAP Binder. However, the oxidative peaks are diminished when RAP binder is blended with PET modified binder. It can be inferred that the amount of oxidative products has been reduced in the PET modified binder with RAP indicating that in combination, it behaves similar to unaged binder.

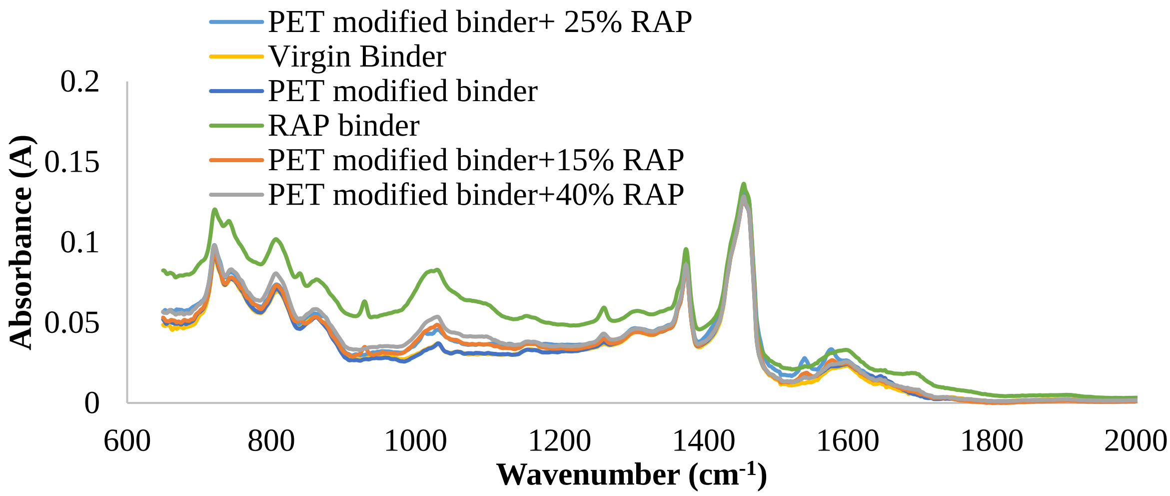


Figure 19. FTIR Spectroscopy Analysis of Mixes

5.7. Fluorescence Microscopy (FM)

In addition to changing the rheological and chemical nature of RAP binder, PET additives also effect the morphology of the asphalt binder. Fluorescence microscope (FM) is a microscopic method, which enables the observation of polymer and additives distribution in asphalt. The mechanism is that the aromatic phase of asphalt is usually the most fluorescent phase, and once polymeric additives absorb aromatics, they become bright when exposed to fluorescence (Polacco et al., 2015). In the conducted test, the wavelength of excitation was in the range 390-

490 nm and the images show that the PET additive has uniformly dispersed inside the asphalt mixture and the dispersed PET is represented by the dotted particles. The different morphologies seen in Figure 20 are a function of the swelling potential of the PET additives, the nature of the virgin asphalt (composition of the maltenes fraction) and the asphalt binder-PET additives compatibility. The different chemical and morphological nature of the PET additive inevitably effects the rheological characteristics of these modified binders.

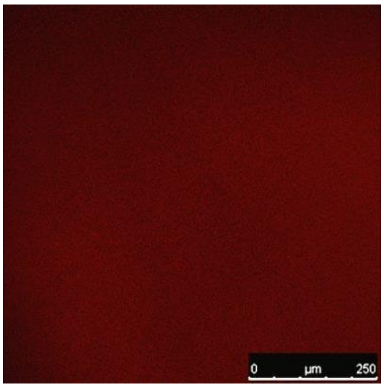


Figure 20. FM of Virgin Binder

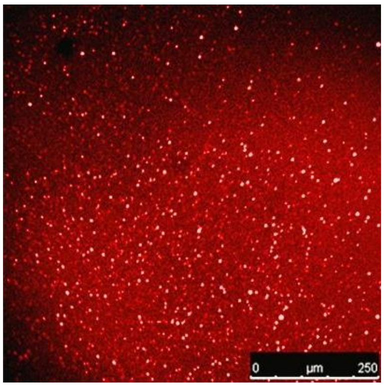


Figure 21. FM of PET Modified Binder

6. Findings and Conclusion

In this study, chemically synthesised PET additive was shown to be suitable to be incorporated in asphalt binders containing RAP; providing better performance to conventional virgin binder. The salient features of the laboratory tests were as follows:

- Based on the anti-stripping tests, the PET additive would be suitable to be incorporated into high RAP mixes as it considerably reduces stripping characteristics of RAP binder.
- The DSR tests indicated that the samples containing RAP binder performed considerably better, improving the fatigue performance at medium temperatures and rutting characteristics at high temperatures.
- The addition of the PET additive also enhanced the low temperature performance of the modified binders with RAP which was then comparable to the virgin binder.
- From the FTIR spectroscopy studies, it was observed that the amount of oxidative products was reduced in the modified binder with RAP indicating the combined mixture behaves similar to unaged binder or that the PET additive could have rejuvenating properties.
- It was established from the fluorescence microscopy images that the mixture of PET modified binder is homogenous which in turn confirms the observed rheological properties of the samples.
- A limitation of this study was that only binder tests were carried out, therefore upcoming research will be focused on conducting mixture studies to ascertain the viability for field tests. In addition, the RAP used was of moderate ageing level. Future tests should also be conducted using various RAP sources to ascertain and quantify the rejuvenating capability of PET additives.

Overall, this study has successfully demonstrated an innovative approach to deal with two waste difficulties: waste plastic and RAP and initiates a competitive technology to meet this recycling challenge. It is anticipated that the combined use of such materials could open a new chapter in the efficient disposal of products currently landfilled and help alleviate the pressure of disposal.

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