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Original Research Article

Properties of Bitumen Modified with Nanoclay/Pet (Polyethylene Terephthalate) Blend

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Abstract

As the world continues to urbanize, the construction of transportation highway continuously requires quality pavement which made transportation engineers and experts focus on improving the performance and life of pavements, to which many studies had searched for better materials or modifications that could improve the properties of bitumen and reduce or even eliminate the development of asphalt pavement failures. To this end, the properties of bitumen modified with blend of PET + nanoclay was conducted. The bitumen was modified with PET (0.5 – 4.0% at 0.5% interval), and nanoclay (1.0 – 8.0% at 1% intervals), while the tests conducted on the materials were oxide composition test, Fourier Transform Infrared Spectroscopy (FTIR) test, penetration test, solubility test, ductility test, flash and fire point test, specific gravity test, softening point test, and viscosity test in accordance with codes and specification. Results from the findings showed that the unmodified bitumen and PET are hydrocarbon materials, while the nanoclay is an inorganic compound and a good reactive pozzolana. More results from the findings showed that the properties of bitumen was improved with addition of PET and nanoclay blend such that the penetration and solubility of bitumen decreases with increase in modifier content, and there was an increase in softening point, flash point, fire point, specific gravity, and viscosity of bitumen as replacement of PET and nanoclay blend increases. Hence, the modifiers (Nanoclay/PET blend) can be used to improve properties of bitumen since they fall within standard and code specification.

Keywords: Bitumen, Polyethylene Terephthalate (PET), Nanoclay, Fourier Transform Infrared Spectroscopy (FTIR), Oxide Composition, Bitumen properties.

1.0 INTRODUCTION

Road infrastructures provide access to industrial zones, agricultural lands, investment, health services, and education. The lack of good roads in particular or the existence of poorly maintained or poor conditions of roads is an obstacle to the development and investment in developing countries (Agbigbe, 2016). It is also pertinent to note that Nigeria as a developing country is facing the problem of flexible pavement failures as result of excessive deformation. As the world continues to urbanize, the construction of transportation roadways constantly requires quality pavement. Due to these demands, transportation experts and engineers focused on improving the performance and life of pavements (Matar, 2017). Many studies and research searching for better materials or modifications that could improve the characteristics of the asphalt mix and reduce or even eliminate the development of asphalt pavement deteriorations (Klinsky *et al*., 2018). Therefore, this study evaluated the properties of bitumen modified with Nanoclay/Polyethylene Terephthalate (PET) blend.

Polyethylene terephthalate (PET), which is a polyester plastic, is one of the most widely used packaging materials for beverages. Due to its excellent transparency, light weight, gas and water barrier properties, impact strength, UV resistance, and unbreakability (compared to a glass bottle), the production and use of PET bottles for beverage packaging has consistently increased worldwide (Benyathiar *et al*., 2022). The global production of plastic bottles has increased from 1.5 million tons per year in 1950 to 245 million tons in 2008, and it has been projected to triple by 2050 (Green-paper, 2013). Plastic is increasingly replacing metals, glass, ceramics and wood in many products. With the above in mind, it is clear that the world and Nigeria, in particular, are already facing enormous challenges in terms of the sustainable disposal of plastic bottles after usage. There is therefore the need to find a way of converting this waste to usable products. Also, the need to utilize readily available cheap materials with the potential of improving bonding in the asphalt pavement to replace the scarce virgin construction materials is of significance in reducing the cost of asphalt production and strength enhancement.

Nanoclay is a component composed of phyllosilicates, which are compounds based on the elements of oxygen, silicon, and other components and are degraded from natural sources and pre-treated chemically. A nano-particle is a miniaturized particle that is measured in nanometers (nm) and is often defined as a particle with at least one dimension that is less than 100 nm. The physics and chemistry of nano-sized particles differ from those of conventional materials, primarily because of the increased surface area-to-volume ratio of nanometer-sized grains, cylinders, plates, and because of the quantum effects resulting from spatial confinement (Yang & Tighe, 2013). The clay Nano particles are the primary materials that could have application in asphalt construction based on a literature review of Nano particles and Nano materials. Carbon Nano tubes (CNT), silica, alumina, magnesium, calcium, and titanium dioxide (TiO_2) Nano particles can also have a significant effect on asphalt performance (Yang & Tighe, 2013). Researches have shown that the additional of nanoclay in asphalt increase the softening point, penetration and ductility (Santagata *et al*., 2012).

Previous studies have shown that, compared to unmodified base binder, the addition of modifier tend to enhance properties of bitumen, depending on the types and dose of the modifier utilized (Abtahi *et al*., 2010; Rubio *et al*., 2012), while other researchers have shown that the physical and mechanical properties of base asphalt binder could also be improved through modification with nanoclay (Galooyak *et al*., 2010; Yao *et al*., 2013). Jahromi and Khodaii (2009) confirmed that nanoclay complexes are highly compatible with organic monomers and polymers which is why nanoclay is used in conjunction with Polyethylene Terephthalate to modify the properties of asphalt binder in this study. To this end, the incorporation of various polymer, micro and nanomaterials into bitumen has been studied increasingly (Ashish *et al*., 2017; Hamedi *et al*., 2015). However, due to availability of numerous types of nanoclays in the market, each one with different properties, there is a need to identify the possibility of using Kaolin based Alkaleri clay obtained from Bauchi State, Nigeria, to improve properties of HMA. The aim of this study will be achieved by characterization of the materials (i.e. pure bitumen, PET, and nanoclay) functional groups; determination of the oxide composition of nanoclay; carry-out particle size analysis to obtain nanoclay from the Alkaleri clay; and determination of the properties of unmodified and modified bitumen.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials used in this study include bitumen, Nanoclay and Polyethylene Terephthalate. The bitumen was obtained at Julius Berger, Nigeria Limited of grade 60/70. The Nanoclay was produced in Ahmadu Bello University, Zaria, Nigeria (Departments of Chemical and Materials and Metallurgical Engineering) from Kaolin based Alkaleri clay obtained from Bauchi State, Nigeria. The Polyethylene Terephthalate waste was obtained from dumps across Zaria, Kaduna State, Nigeria, and was used for the modification of bitumen.

2.2 Methods

2.2.1 Preparation of PET and Nanoclay

2.2.1.1 Preparation of PET

The polyethylene terephthalate (PET) bottles were washed, dried and grinded by mechanical means. After shredding the PET was grinded and sieved and those that passed through sieve 2.36 mm were used.

2.2.1.2 Preparation on Nanoclay

Precipitation method was used to obtain nanoclay from the clay. After obtaining the unprocessed clay, it was ball milled for 100 hours and soaked in water for 24 hours, after which it was wet sieved through sieve 0.1um, and the water was decant and oven dried for 24 hours, and it was pulverised for 4-5 hours. The particle size analysis (PSA) was done in accordance with (BS-3406-1, 1986) to obtain the nanoclay particles.

2.2.2 Oxide Composition Test

Oxide composition test was carried on PET and the Nanoclay. The Energy dispersive spectrometer designed for detection and measurement of elements in a sample according to ASTM-C618 (2019). The test was conducted at Chemistry Department of Ahmadu Bello University, Zaria, Nigeria.

2.2.3 Fourier Transform Infrared Spectroscopy (FTIR) Test

The FTIR test was conducted on the materials (bitumen, PET, and nanoclay) using Thermo Fisher spectrometer at multi-user science research laboratory of Ahmadu Bello University, Zaria, Nigeria. The spectra were recorded from 4000–500 cm^{-1} at a resolution of 4 cm^{-1} averaging 76 scans for each measurement. For each sample, 16 trials were performed for analysis. To avoid the variability in spectra induced by sample thickness, indices were calculated for each sample, and repeatability limit was calculated for the indices rather than for the whole spectra.

2.2.4 Test on bitumen (Modified and Unmodified)

- Penetration test (ASTM-D5/D5M-20, 2020),
- Solubility test (ASTM-D2042-15, 2015),
- Ductility test (ASTM-D113-17, 2017),
- Flash and fire point test (ASTM-D92-18, 2018),
- Specific gravity test (ASTM-D70/D70M-21, 2021)
- Softening point (ASTM-D36/D36M-14, 2014).
- Viscosity (ASTM-D4402/D4402M, 2015).

2.2.5 Modification of Bitumen

During the modification process of the bitumen binder, both additives were added to the bitumen before introducing the mixture into the mineral aggregates. The percentages of the nanoclay ranged from 1.0 – 8.0% at 1.0% intervals, while the PET ranged from 0.5 – 4.0% at 0.5% interval by weight to the bitumen binder as shown in Table 1.

Table 1: Modification of Bitumen with Pet and Nano Clay

Mass of Bitumen (g)	PET (%)	Mass of PET (g)	Nanoclay (%)	Mass of Nanoclay (g)	Mass of PET and Nano Clay (g)
160	0.5	0.8	1.0	1.6	2.4
160	1.0	1.6	2.0	3.2	4.8
160	1.5	2.4	3.0	4.8	7.2
160	2.0	3.2	4.0	6.4	9.6
160	2.5	4.0	5.0	8.0	12.0
160	3.0	4.8	6.0	9.6	14.4
160	3.5	5.6	7.0	11.2	16.8
160	4.0	6.4	8.0	12.8	19.2

3.0 RESULTS AND DISCUSSION

3.1 Properties of Unmodified Bitumen

The properties of unmodified bitumen was determined, and the properties evaluated were the penetration, ductility, softening point, flash point, fire point, specific gravity, solubility, and viscosity of the bitumen. Table 2 shows the properties of the unmodified bitumen.

Table 2: Results of Unmodified Bitumen Properties

Test conducted	Code	Result	Specifications	Remark
Penetration (mm)	ASTM-D5/D5M-20 (2020)	67.0	60-70	OK
Softening point (^{0}C)	ASTM-D36/D36M-14 (2014)	49.9	46-56	OK
Flash point (^{0}C)	ASTM-D92-18 (2018)	257	<302	OK
Fire point (^{0}C)	ASTM-D92-18 (2018)	290	<310	OK
Ductility (cm)	ASTM-D113-17 (2017)	110	Min. 100	OK
Specific gravity	ASTM-D70/D70M-21 (2021)	0.99	0.98-1.02	OK
Solubility	ASTM-D2042-15 (2015)	100	Min. 99	OK
Viscosity	ASTM-D4402/D4402M (2015)	157	Min. 120	OK

The results from Table 2 of the unmodified bitumen showed that the penetration is 67.0 which is within the range for grade 60/70. Results of other physical properties such as softening point (49.9^{0}C), flash point (257^{0}C), fire point (290^{0}C), ductility (110cm), specific gravity (0.99), solubility (100%), and viscosity (157) are within the range specified by American Society for Testing and Materials (ASTM) codes as shown in Table 2. Therefore, the bitumen is a 60/70 penetration grade bitumen and is suitable for the production of asphalt since all properties evaluated falls within code specification.

3.2 Material Characterization and Oxide Composition

3.2.1 Material Characterization

Fourier Transformation Infrared Radiation (FTIR) was used to characterize the materials (i.e. bitumen, PET, and Nano clay) based on infrared radiation transmission and the material absorption to identify the functional element groups in the materials, and the results are presented in Figures 1–3.

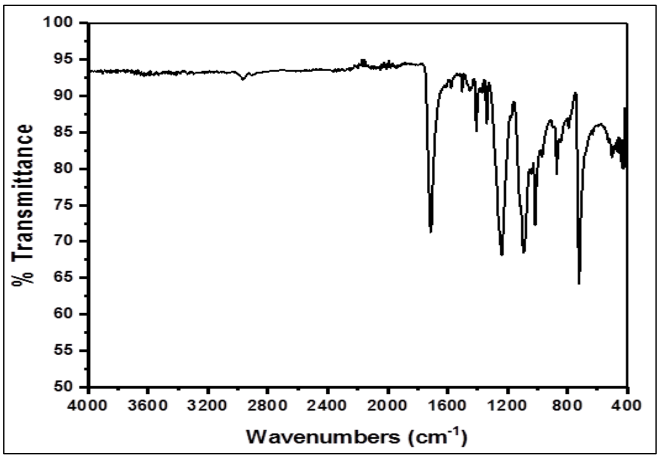

Figure 1: FTIR of Bitumen Sample

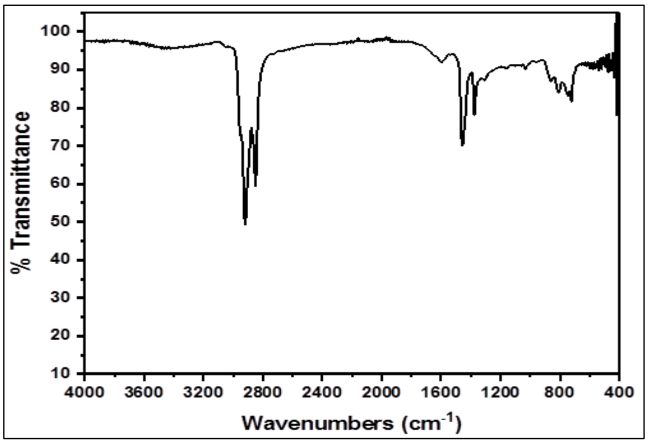

Figure 2: FTIR of PET Sample

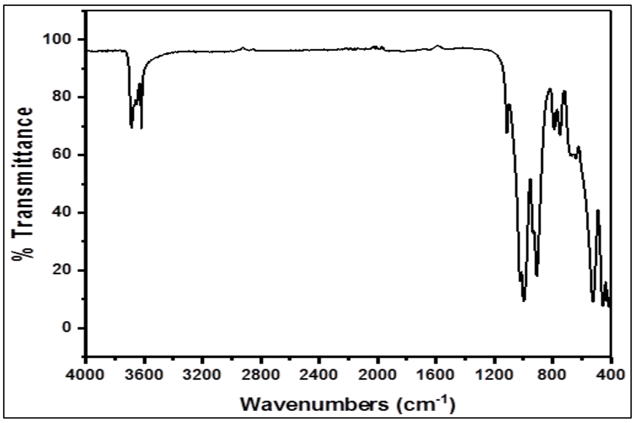

Figure 3: FTIR of Nano clay Sample

For the bitumen sample in Figure 1, the sharp peak corresponding to 1712cm^{-1} suggests the presence of carbon double bond (C=O stretch), the peaks at 1399, 1408, 1505, and 1578cm^{-1} corresponds to C=C vibration in aromatics, while the peaks at 1016, 1093, 1239cm^{-1} indicates an C-H vibration. At the fingerprint region, the peak at 723 indicates the presence of molecules with more than four carbon atoms in a row. The prominence of these peaks is expected as bitumen is composed of hydrocarbons with about 81% of carbon and 10% of hydrogen (Nivitha *et al*., 2016).

The FTIR analysis of the PET shown in Figure 2 shows that there exist two short sharp peak of wavenumber 1376 cm^{-1} and 1456 cm^{-1} which indicates the presence of C-H functional group, there also exist two medium sharp peak of wavenumber 2850cm^{-1} and 2920cm^{-1} which indicates the presence of Sp^3 C-H functional group. Although, the prominence of these peaks is as high as the bitumen, it also comprises of C-H bonding i.e. hydrocarbon. Hence the PET exhibits some similar properties of the bitumen.

The FTIR analysis of the Nano clay material shown in Figure 3 shows that there exist a short strong sharp peak of wavenumber 1025 cm^{-1} which is due to Si-O planar stretching common with kaolinite. Also at the fingerprint region (400-1000cm^{1-}), the bands at 407, 457, and 524cm^{-1} are also indicating Si-O bending vibration, while the peaks at 909, and 999cm^{-1} are indicative of Al-O-Si inner surface vibration (Davarcioğlu & Çiftçi, 2009), which is also an indication of impurities in the clay ample a reported by (Thomas, 1989). Hence, from the FTIR analysis, the nanoclay sample is a kaolinite inorganic material with some level of impurities.

3.2.2 Materials Oxide Composition

Energy Dispersive X-Ray Fluorescence (EDXRF) test was conducted on the Nano clay used in this study to determine the chemical composition of the materials and the result are presented in Table 3.

Table 3: Oxide Composition of Nanoclay

Oxides Composition	Nanoclay (%)
Na_2O	
MgO	4.470
Al_2O_3	35.210
SiO_2	44.287
P_2O_5	0.1106
SO_3	0.0876
Cl	0.0406
K_2O	1.5254
CaO	0.0473
TiO_2	0.04091
Cr_2O_3	0.00057
Mn_2O_3	0.02062
Fe_2O_3	0.3631
ZnO	0.00559
SrO	0.0373

The result from Table 3 shows the oxide composition of nano clay used in this study, and from the table, the combination of SiO_2, Al_2O_3 and Fe_2O_3 of the nano clay is approximately 79.86% which is above 70% as specified in ASTM-C618 (2019), and is an indication that the nano clay used in this study is a good reactive pozzolana. However, since SiO_2 is greater than 39.9 and less than 54.9, with an SO_3 value less than 5%, the nano clay material is a Class F pozzolan (ASTM-C618, 2019). Also, the presence of siliceous and aluminous material in the nano clay powder indicates that in finely grounded form, it can react with calcium hydroxide to form calcium silicates hydrate (CSH) which is a strength forming product (ASTM-C618, 2019).

3.2.3 Particle Size Analysis

To make sure the clay material is in the nanoscale, particle size analysis was conducted for unprocessed and processed alkaleri clay as shown Figures 4 and 5 respectively.

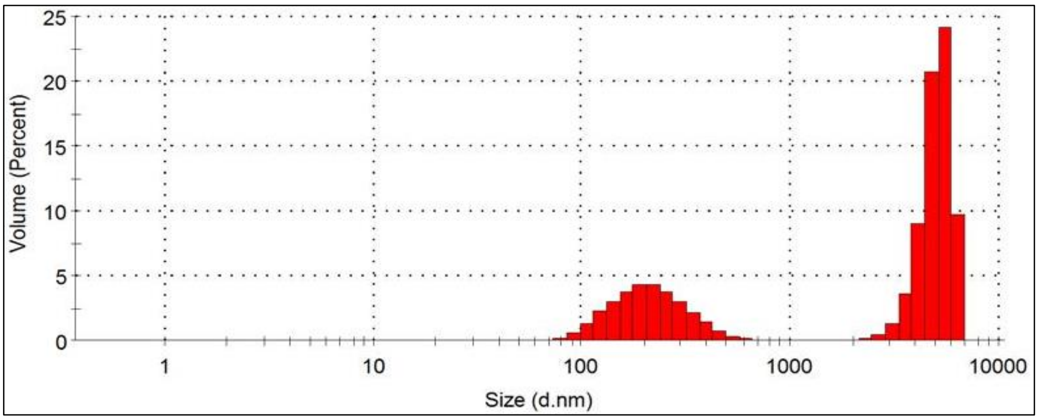

Figure 4: Unprocessed Alkaleri Clay

Figure 4 shows that the unprocessed nanoclay particle size ranged from 100nm (1×10^{-7}) to approximately 7000nm (7×10^{-6}), with the majority of the particle size falling within 3000nm-7000nm. However, since majority of the particle size is not between 1-100nm, it's not a suitable nano-material (Yang & Tighe, 2013).

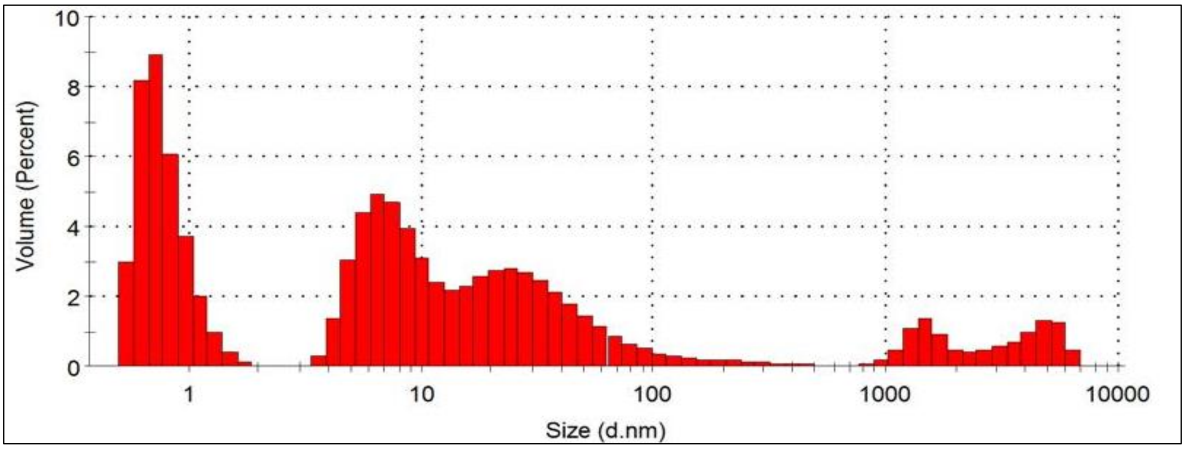

Figure 5: Processed Alkaleri Clay

Result from Figure 5 shows that after processing of Alkaleri clay, the majority of the particle size ranged from approximately 1nm (1 x 10^{-9}) to 100nm (1 x 10^{-7}) with about 90% of the nanoclay by volume having a particle size between 1-2nm. Since the processed clay have most dimension between 1nm-100nm, it qualifies to be a nanoclay-material (Yang & Tighe, 2013).

3.3 Properties of Bitumen Modified with PET and Nanoclay

The properties of bitumen modified with PET and nanoclay at various percentages was determined and the results presented in Figures 6–13.

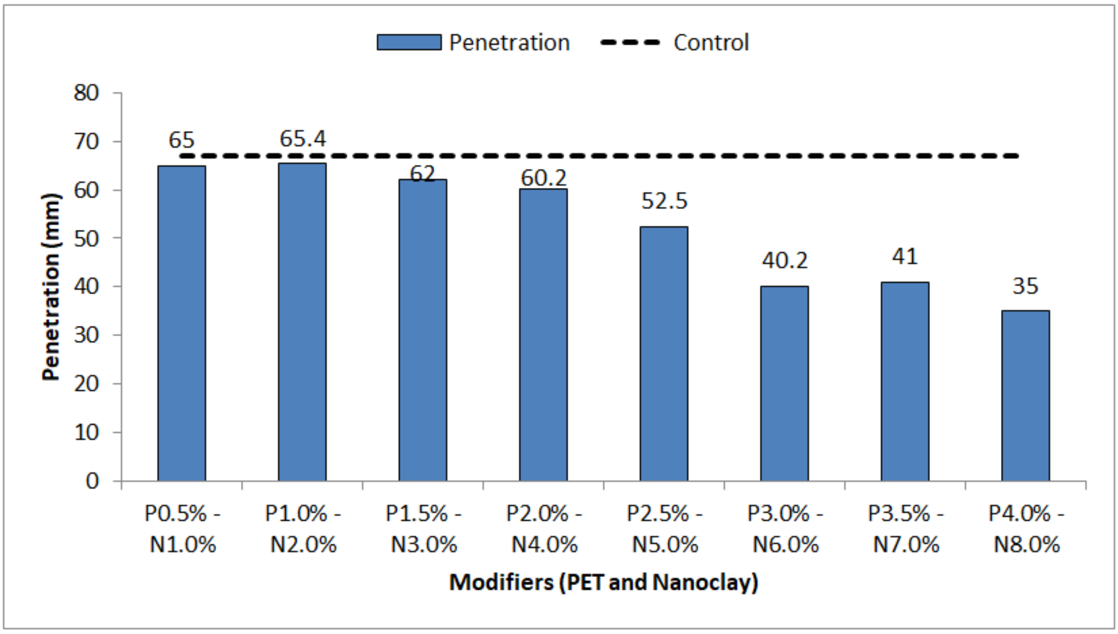

Figure 6: Penetration of Bitumen Modified with PET and Nanoclay

The result from Figure 6 shows that the penetration of bitumen modified with blend of PET + nanoclay reduces as replacement increases with an optimum penetration value at P0.5% - N1.0% (65.0mm) and P1.0% - N2.0% (65.4mm). The reduction in penetration of the modified bitumen is as a result of the PET absorbing low molecular components of the bitumen, provoking a swelling in their structures and a movement restriction between the molecules inside the asphalt, which then result in a decrease in the penetration and is in agreement with finding of Ahmad and Ayob (2016); Ahmedzade *et al*., (2017); Fang *et al*., (2011); Jan *et al*., (2018); Murana *et al*., (2020); Olukanni (2018). It can also be as a result of the nanoclay large surface area and its stiffening effect by forming bond chains within the binder which is also in agreement with the findings of El-Shafie *et al*., (2012); Golestani *et al*., (2012); Jahromi and Khodaii (2009).

Hence, as nanoclay finer particle keeps forming bond with the PET and binder, it contributes to further stiffening of the bitumen and reduced penetration. The outcome of these findings shows that the use of nanoclay and PET can be used to modify bitumen to lower penetration grade (35-70mm) with reduction in penetration of bitumen as modifier content increase. The engineering implication of the low penetration of the modified bitumen samples is an indication that it has a higher resistance to temperature changes and heavy traffic loads, which will lead to longer lifespan of the pavement, and lower maintenance costs. Low bitumen penetration improves skid resistance which enhances safety on roads, and it also reduces water infiltration into the pavement structure, which enhances pavement durability.

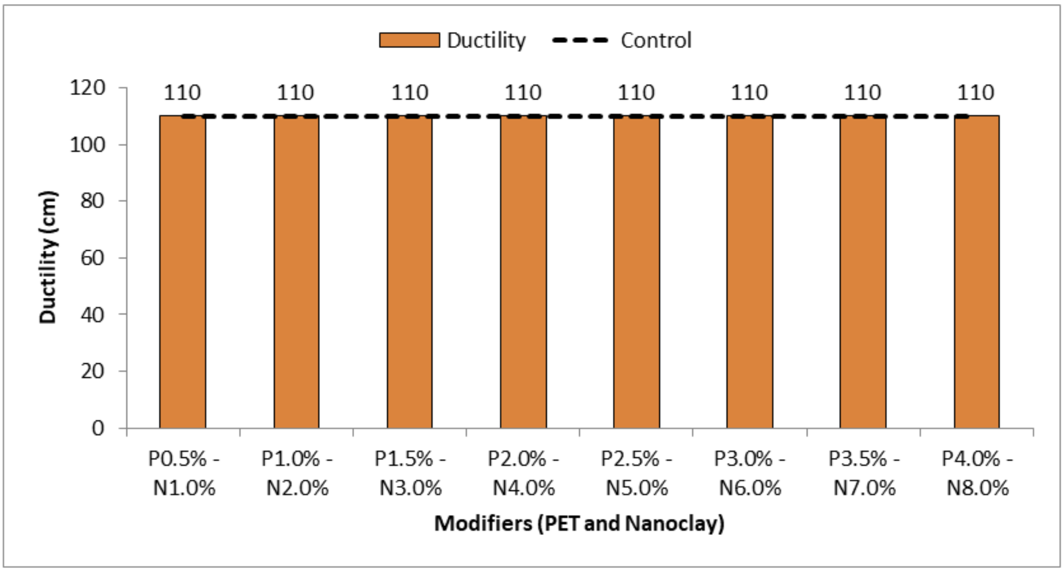

Figure 7: Ductility of Bitumen Modified with PET and Nanoclay

The result from Figure 7 shows that ductility of bitumen modified with blend of PET and nanoclay maintained a constant value of 110cm with the control bitumen (110cm), which is an indication of good adhesive properties and good performance of the bitumen in service. However, the outcome of the findings shows that ductility of modified and unmodified bitumen sample met the requirement (min 110cm) in accordance with ASTM-D113-17 (2017) specification.

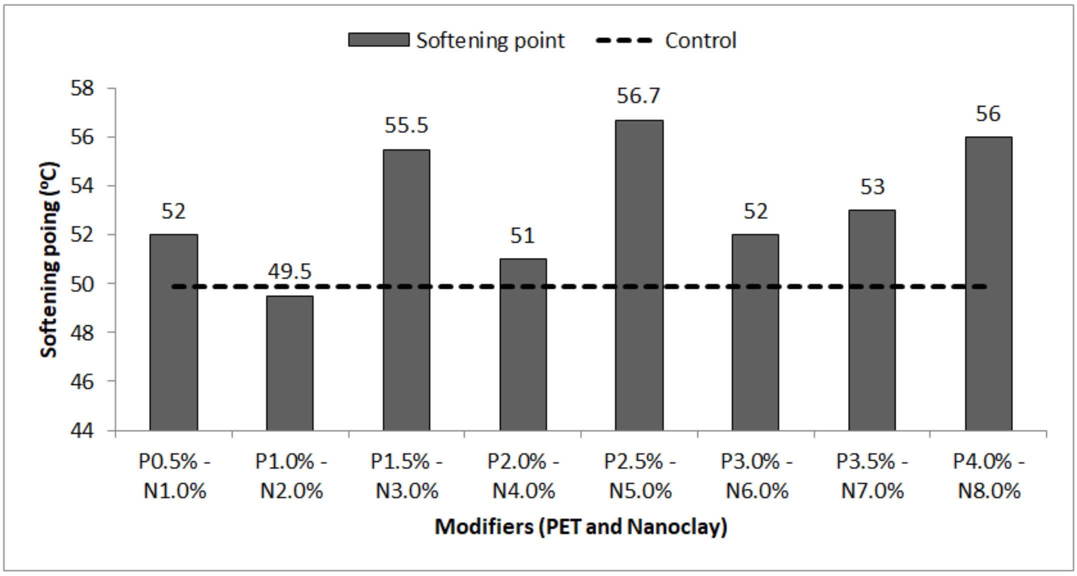

Figure 8: Softening Point of Bitumen Modified with PET and Nanoclay

The result from Figure 8 shows that the softening point of bitumen modified with PET and nanoclay follows an irregular pattern and are all higher than the control bitumen except for bitumen modified with P1.0% – N2.0%. However, the softening point value of all the bitumen were within 46 – 56ºC specified by ASTM-D36/D36M-14 (2014) except for bitumen modified with P2.5% - N6.0% with a value of 56.7ºC. The increase in the softening point can be due to the internal structure of the polymer and the softening point have relationship with bitumen penetration since penetration is a property that is highly related to viscosity, and viscosity is a direct influencing agent of the softening point property (Thom, 2008). Hence, as penetration decrease, softening point increase. More also, The increase in the softening point is partly due to Nano clay modification improving the rheological properties of the binder by increasing the stiffness of bitumen and decreasing the phase angle (improves elasticity) compared to conventional bitumen (Jahromi and Khodaii, 2009). The result is in accordance to the findings of Abdullah *et al*., (2016); Blom *et al*., (2017); El-Shafie *et al*., (2012); Golestani *et al*., (2012); Santagata *et al*., (2012).

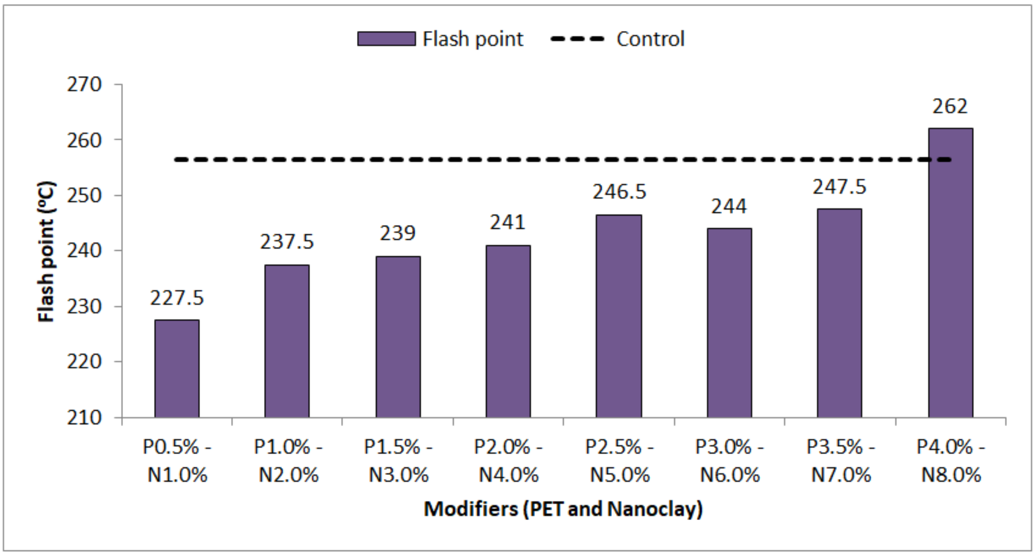

Figure 9: Flash Point of Bitumen Modified with PET and Nanoclay

The result from Figure 9 shows that the flash point of bitumen modified with PET and nanoclay all fell within standard specification (<302^{0}C) ASTM-D92-18 (2018) and are all less than the control bitumen except for bitumen modified with P4.0 – N8.0%. This is because nanoclay enhances the flame retardancy and thermal stability of bituminous binders since nanoclay layers have high aspect ratio and are dispersed at nanoscale in bituminous binders (Wu *et al*., 2008). Hence, the modified bitumen has lower flammability compared to the unmodified bitumen and can be used in regions with high temperatures with less hazard in transportation, storage, and handling.

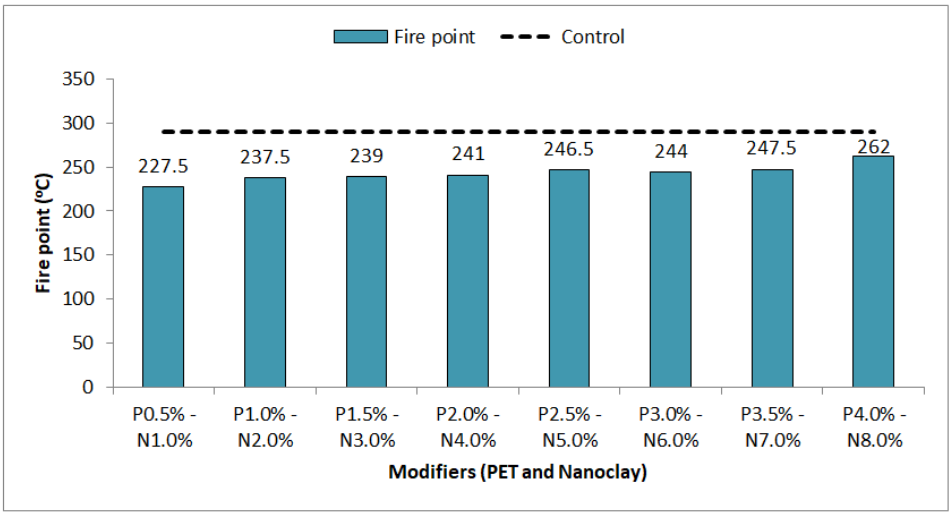

Figure 10: Fire Point of Bitumen Modified with PET and Nanoclay

The result from Figure 10 shows that the fire point of modified and unmodified bitumen all fell within standard specification (<310^{0}C) ASTM-D92-18 (2018). Also, all the modified bitumen fire point values were less than the unmodified bitumen (control bitumen). This is due to the strong interfacial interaction between bitumen and nanoclay. Nanoclay protects bitumen against thermo-oxidation, and this property is derived from "labyrinth" effect of silicate layers in nano clay. Nanoclay can catalyze char-forming reactions to form a charred residue which combined and intercalates with silicate layers to provide a sort of carbonaceous silicate structure on the surface, which also significantly delays the escape of volatile products and retards the penetration of oxygen (Wu *et al*., 2008).

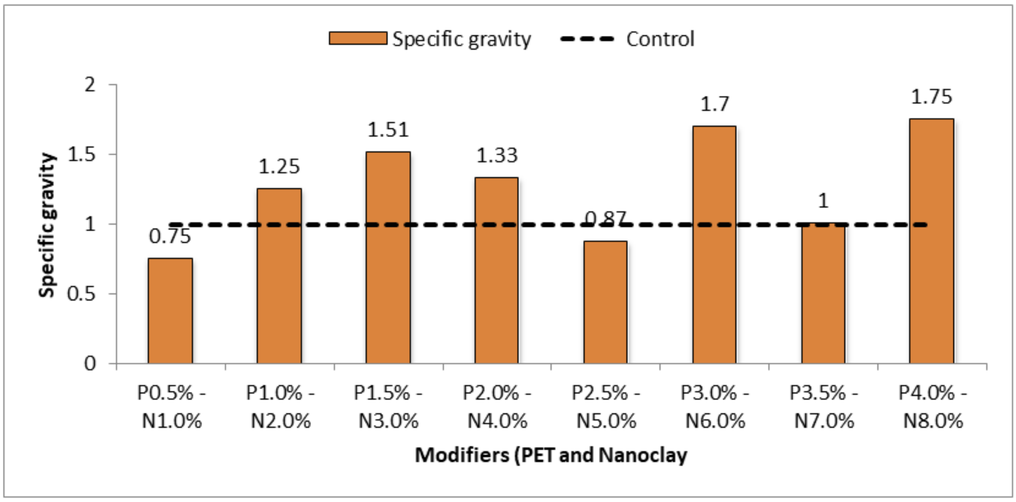

Figure 11: Specific Gravity of Bitumen Modified with PET and Nanoclay

The result from Figure 11 shows that PET and nanoclay modified bitumen had specific gravity greater than the control bitumen except for bitumen modified with P0.5 – N1.0%, and P2.5 – N5.0%. Also, most of the modified bitumen had specific gravity values outside the recommended range of 0.97 – 1.06 ASTM-D70/D70M-21 (2021), except for bitumen modified with P0.5 – N1.0%, P2.5 – 5.0%, and P3.5 – N7.0%. The outcome of this finding showed that the low specific gravity of the modified bitumen will lead to lower concentration of the bituminous materials, resulting in weaker pavements that deteriorate rapidly under heavy traffic conditions or harsh weather conditions. On the other hand, the high specific gravity of the modified bitumen that falls within the specified limit, means the modified bitumen have better adhesion properties, making it more durable for mixing with aggregates in asphalt concrete paving.

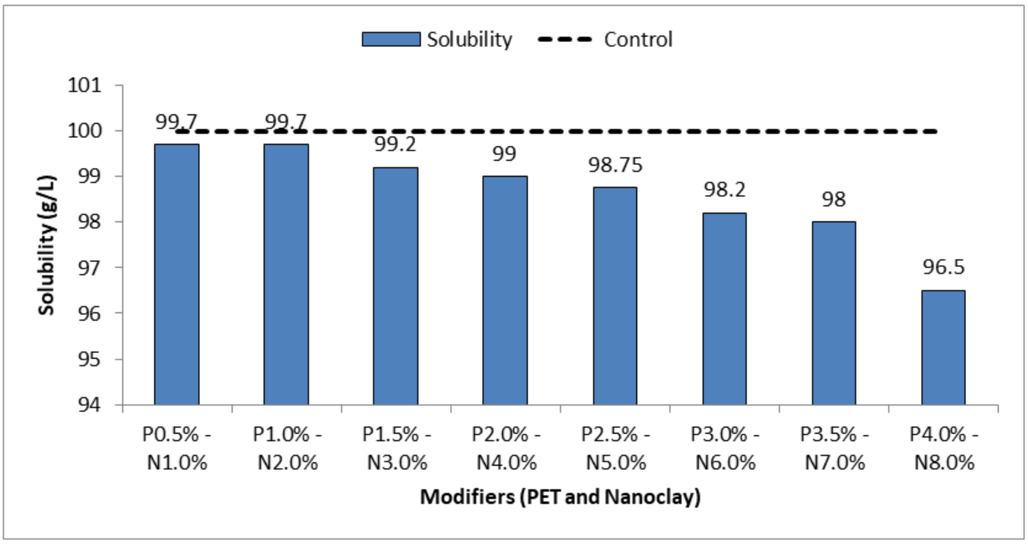

Figure 12: Solubility of Bitumen Modified with PET and Nanoclay

The result from Figure 12 shows that solubility of bitumen modified with PET and nanoclay are all lower than the control, and the solubility also reduces as modifier replacement content increases. Also, bitumen modified with P2.5 – N5.0% to P4.0 – N8.0% had values lower than 99 specified by ASTM-D2042-15 (2015) which is an indication that they might contain some impurities which makes it less soluble. However, the FTIR analysis from Figure 1.0 – 3.0 already indicated that the nanoclay used in this study contains some impurities due to the Al-O-Si inner surface vibration (Davarcioğlu & Çiftçi, 2009). Hence, it can contribute to the level of impurity in the PET and nanoclay modified bitumen. The implication of the outcome of this findings showed that the low solubility of the modified bitumen is an indication these blend of bitumen can lead to poor pavement performance due to inadequate bonding, that will lead to early cracking and rutting, since proper bitumen solubility ensures that only the desirable amount of bitumen remains in the roadway's surface layer, in order preventing excessive bleeding or rutting. Hence, low soluble bitumen will lead to decreased adhesive properties resulting in pavement failure.

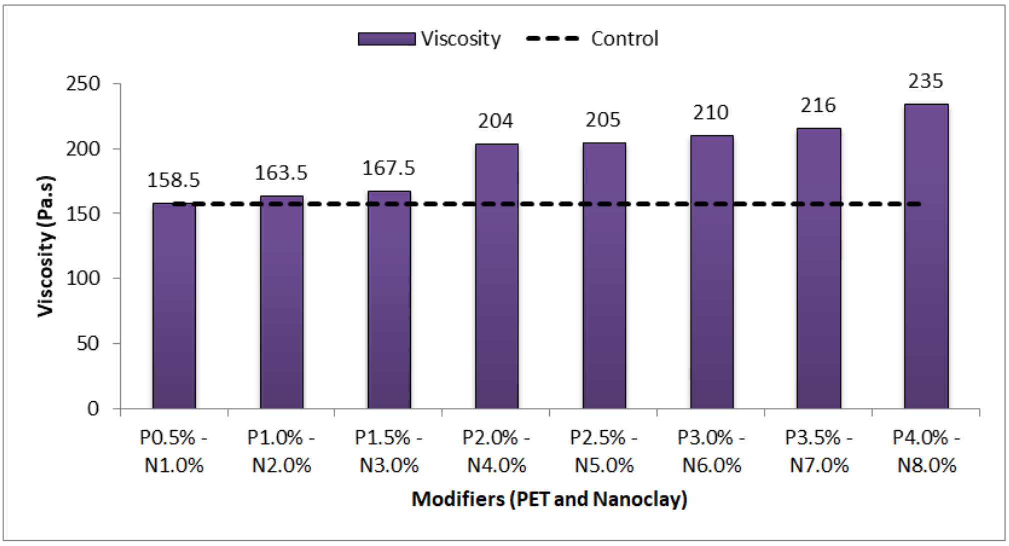

Figure 13: Viscosity of Bitumen Modified with PET and Nanoclay

The result from Figure 13 shows that viscosity of bitumen modified with PET and nanoclay increases as replacement increases and falls within the minimum value of 120 Pa.s specified by ASTM-D4402/D4402M (2015). This reduction in viscosity can be attributed to efficient dispersion of the nanoclay, due to the large surface area and the stiffening effect of the nano clay (El-Shafie *et al*., 2012), it can also be attributed to the interaction between plastic and bitumen particles (Lin *et al*., 2019), or due to the creation of a three-dimensional network within the bitumen. This network creates an internal movement resistance and thus an increase in the viscosity (Al-Abdul Wahhab *et al*., 2017; Farahani *et al*., 2017). The outcome of these findings is an indication that the high viscosity of the modified bitumen has a more adhesive property that have the ability to prevent water from penetrating into the pavement layers, thus reducing the risk of damage caused by freeze-thaw cycles or other weather conditions. The high viscosity of the modified bitumen also indicates that they can be used in regions with high temperature since it has improved resistance to temperature. High viscous bitumen when also used in HMA can create a strong bond with the aggregate materials, making it resistant to wear and tear caused by heavy traffic and adverse weather conditions.

4.0 CONCLUSION

This study was conducted to determine the properties of bitumen modified with blend of PET and nanoclay and at the end of the findings, it was observed that the nanoclay used in the study is a good reactive pozzolana that can react with calcium hydroxide to form calcium silicates hydrate (CSH) which is a strength forming agent. The FTIR analysis identified C=O, C=C and C-H functional groups in the unmodified bitumen and PET which makes it hydrocarbon materials, and the FTIR identified Si-O, and Al-O-Si functional group in the nanoclay which makes it an inorganic compound. The particle size of the unprocessed nanoclay is in micrometres (10^{-6}), while particle size of the processed nanoclay is in nanometers (10^{-9}) with majority of the clay ranging between 1 - 100 nanometer, which makes is a suitable nanoclay material. Finally, results from the findings showed that the modified bitumen (blend of PET and nanoclay) compared to the unmodified bitumen showed a decrease in penetration, and solubility, with an increase in softening point, flash point, fire point, specific gravity, and viscosity as replacement level increases. Hence, the modifiers (Nanoclay/PET blend) is a good material that should be used to improve properties of bitumen, since the properties of the modified bitumen improved and mostly falls within standard and code specification.

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