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Utilization of Indigenous Sourced *Borassus aethiopum* Fruit Cap Fibre in Enhancing the Physical, Mechanical and Thermal Properties of Waste LDPE Composites

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Abstract: In this work, the physical, mechanical, dynamic mechanical and morphological properties of recycled low density polyethylene were enhanced with the incorporation of *Borassus aethiopum* (BA) fibres and $BaSO_4$ powder as nucleating agent. The materials were compounded on a Two Roll Mill and compression moulding machines. Properties such as density, water absorption, tensile strength, elastic modulus, flexural strength and hardness values were evaluated using standard procedures. The materials were further subjected to dynamic mechanical properties test in order to evaluate the influence of BAP fibre addition on the storage modulus, loss modulus and damping parameter waste low density polyethylene (rLDPE). Tensile strength of 55.50 MPa, stiffness of 3.89 GPa, flexural strength of 65.80 MPa and hardness value of 44.88 Shores at 24 % weight fraction of reinforcement with corresponding minimal water absorption of 6.65 % after 240-hrs immersion period at room temperature. These are all higher than that of the unreinforced rLDPE (control sample). Similarly, dynamic mechanical properties results indicated that the storage modulus and load bearing capability of were found to be maximum (1.6 GPa) for a composite with 24 % weight of BAP fibre compared (to 160 MPa of the unreinforced rLDPE). Similarly, as the frequency was increased, the composite's response becomes more elastic, meaning it stores more energy rather than dissipating it which account for the composite decreased loss modulus. Damping parameters of the composite materials also indicate a gradual decrease with the amount of BAP incorporated from 0.288 (of unreinforced rLDPE) to 0.08 (of BAP-rLDPE) at 24 % which suggests that the sample with 24 % BAP has the best load bearing capability of all the composites. These results indicate that incorporation of BA fibre into rLDPE improved its physical, mechanical, dynamic mechanical and morphological properties.

1. Introduction

Plastic use is fast expanding year after year, and this trend is expected to continue due to the remarkable versatility of plastic materials' features, which allow them to be used for a wide range of purposes resulting in a large volume of waste (Jacob, 2023). It is projected that the amount of plastic waste in municipal solid garbage will increase at a pace of two times every decade. This rise is closely linked to the rapid changes in population, urbanization, and developmental activities and lifestyle. Studies have shown that waste plastics can withstand 450–500 years at Earth's surface before degrading (Thirugnanasambantham *et al.*, 2017; Shinde *et al.*, 2018). Adopting a recycling procedure in the form of composites is one approach to appropriately handle the tons of plastic garbage that need to be disposed of, and recycling also helps to maintain a cleaner environment. Due to the severe limitations controlling their disposal and the mounting environmental difficulties brought on by waste plastics, researchers are becoming more and more driven to develop new innovative products in the form of composites (Lendvai *et al.*, 2024; Jacob *et al.*, 2024).

Polymer composites are widely employed in industries such as transportation, aircraft, and packaging due to their superior corrosion resistance, low density, translucency, and stiffness (Jacob, 2019; Shah *et al.*, 2023; Jacob, 2023 and Kumar *et al.*, 2024; Afroz *et al.*, 2024; Kane *et al.*, 2024). Natural fibres enhance the properties of the constituent polymer composite by improving their mechanical, thermal and electrical properties. These natural fibres are safe, biodegradable, easily accessible, and lightweight. As a result, they are commonly employed to strengthen plastics in the form of composites. Natural fibres are a viable alternative to synthetic reinforcements due to their broad availability and versatility in surface treatments (Akartasse, *et al.*, 2022; Aaddouz, *et al.*, 2023). These composites are utilised in the building and automotive industries (Rauniyar, *et al.*, 2024).

In addition to mechanical, physical and morphological properties of polymer composites; the thermal characteristics of a polymer composite are quite important, particularly with regard to the production parameters and areas of application (Nanni, *et al.*, 2021). One of the most crucial techniques for researching the thermal characteristics of polymers is dynamic mechanical analysis (DMA) (Jacob, 2023). Robust research is needed to give better performance qualities for these continuously expanding natural fibre-plastic composites to meet the expectations of end-user applications (Jacob and Yusuf, 2023). Researchers are also more interested in maximizing renewable resources, such as natural fibers, in producing composites due to the necessity to lessen our over-reliance on fossil fuels and synthetic fibres (Jacob and Mamza, 2021).

This study, therefore, tends to investigate safer methods of plastic waste disposal by exploring environmentally safe and innovative methods, one of which is composite development, to promote a sustainable environment.

2. Methodology

2.1 Sourcing and preparation of low density polyethylene wastes and Borassus aethiopum fibre

Low-density polyethylene waste was gathered from landfills. These samples were water-washed to remove dirt, dried, and shredded into the smaller particles that make up the polymer matrix. The Borassus aethiopum fibre (also known as African fan palm) fruit cap utilized as reinforcement was obtained locally from Hong Local Government Area, Adamawa State, Nigeria. It was sun-dried, ground, and sieved to a size of 100 µm.

2.2 Experiments

2.2.1 Composite development

The materials were compounded at 160 °C on a Two Roll Mill according to the findings of (Jacob *et al.*, 2018a and Jacob *et al.*, 2019; Jacob *et al.*, 2024), the materials compositions ranged from 4-24 % and (96, 92, 88, 80 and 76 %) of BAP and rLDPE respectively with 5 g of $BaSO_4$ powder added as the nucleating agent. After that, the samples were cured for ten minutes using a hydraulic press set to 170 °C and 3 Pa of compression pressure. The obtained samples were machined and chilled before going through characterisation testing.

2.3 Composite characterisation

2.3.1 Mechanical properties test

2.3.1.1 Tensile test

The ASTM D 638 (2014) suggested procedure was used to determine the samples' tensile strength results. Following the samples' dumbbell-shaped machining, they were put in the computerized Instron Universal Tensile Testing Equipment 3369 model, which assessed the samples' elastic modulus and tensile strength.

2.3.1.2 Flexural strength

In accordance with ASTM D790 (2015), flexural strength was determined by utilizing a universal testing machine and a three-point bending technique. The strain rate was 5 mm/min, and the spans were 40 mm apart. Equation (1) was used to calculate the flexural strength (MPa):

$$\sigma = \frac{3Pl}{2bt^2} \quad (1)$$

l = length of specimen span between support (mm)
P = maximum deflection force (N)
b = width of specimen (mm)
t = thickness of specimen (mm)

2.3.1.3 Hardness test

According to Jacob *et al*. (2018a), the relative resistance of a composite's surface to indentation by an indenter of a certain size under a given load is the basis for the hardness test of the material. Durometer Shore A was used to measure the shore hardness values of samples measuring 30 mm by 30 mm by 5 mm. The sample underwent five measurements at various locations, and the average of the results was used to determine the sample's hardness.

2.3.2 Physical Properties Test

2.3.2.1 Density

The density of the composites was ascertained by measuring the mass and volume of the sample in accordance with the research of Jacob (2019).

2.3.2.2 Water Absorption test

The water absorption test was carried out utilising the ASTM D570 (2010) methodology. The test sample was an oven-dried specimen measuring 76 x 25 x 5 mm submerged in water at room temperature for 24 hours. After a 24-hour immersion period, the specimens were removed and cleaned with a lint-free cloth before being weighed again using a Sartorius ED 224S digital analytical scale. Both the dried weight before and after weight immersion (W_2) were recorded. Jacob (2019) reported a similar technique. The moisture uptake in the composite was determined using equation (2):

$$W = \frac{W_{final} - W_{initial}}{W_{final}} (\%) \quad (2)$$

2.3.3 Thermal Properties

2.3.3.1 Dynamic mechanical analysis

A DMA 242 E machine was used to execute DMA in compliance with ASTM D7028 (2015). The test parameters - storage modulus (E'), loss modulus (E"), and tangent of delta (Tan ∂) - were initially built up using a personal computer and the proteus software. The instrument configuration included the furnace temperature range (30-110 °C), dynamic load (4 N), frequency range (1-10 Hz), sample holder (3-point bending), and heating rate (3 K/min). Each test produced a sample measuring 60 x 12 x 5 mm. The test specimens were loaded into the equipment using three-point bending before being locked into the furnace.

3. *Results and Discussion*

3.1 Mechanical Properties

3.1.1 Effect of BA powder on the tensile strength of recycled LDPE composites

Figure 3 depicts the tensile strength of BAP-rLDPE composites with increasing proportion of the fibres. Tensile strength improves from 5 % to 20 %, then declines at 25 %. The rise in tensile strength with weight percentage of fibre could be attributed to weakening of the constituent composition's interfacial attraction as the proportion of rLDPE decreases with increasing weight fraction of reinforcement. *Other researchers have observed similar observations* (Jacob *et al*., 2018b; Jacob and Mamza, 2021).

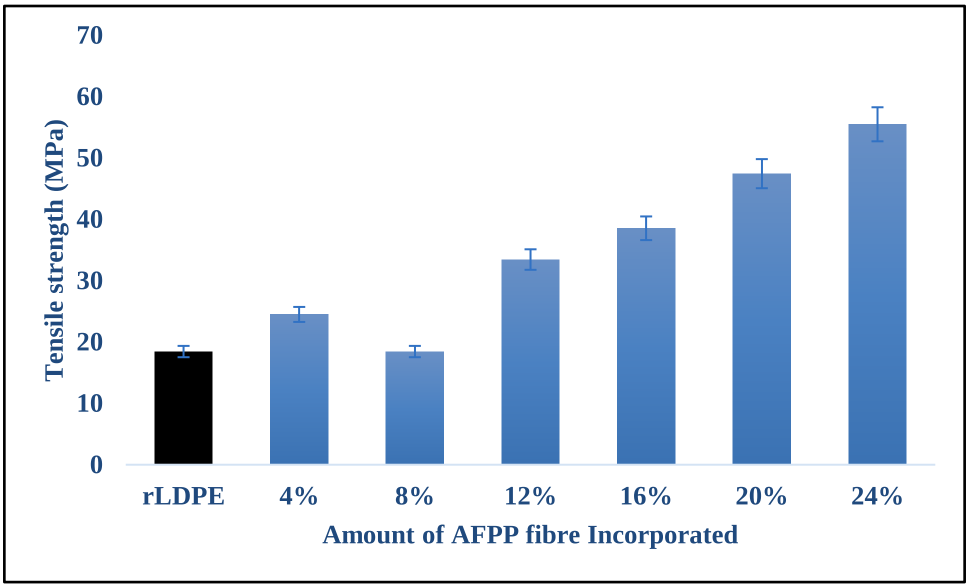


Figure 1: Effect of BA powder on the tensile strength of recycled LDPE composites

3.1.2 Effect of BA powder on the elastic modulus of recycled LDPE composites

Figure 2 shows the elastic modulus (stiffness) of the BAP-rLDPE composite as a function of reinforcement weight. A comparable rise in stiffness with weight percentage of rLDPE was observed. The composites' elastic modulus increases from 1.23 GPa (rLDPE) to 2.1 GPa (at 24 % BAP). This could be attributed to improved interaction between rLDPE and the integrated BAP fiber. These findings are consistent with those of other researchers (Khalaf, 2015; Jacob *et al*., 2019b; Jacob and Mamza, 2021).

3.1.3 Effect of BA powder on the flexural strength of recycled LDPE composites

Figure 3 depicts the composites' flexural strength in relation to their weight percentage of BAP fibres. The flexural strength of *Borassus aethiopum* powder reinforced rLDPE composites increases with the weight fraction of reinforcement and thereafter drops, reaching 68 MPa at 20 % wt reinforcement. This indicates better contact and stress transfer between the rLDPE and BAP particles. It was discovered that increasing the weight fraction of reinforcement (at 25 %) reduces the flexural strength value due to poor fibre-matrix adhesion (Jacob and Shinggu, 2021).

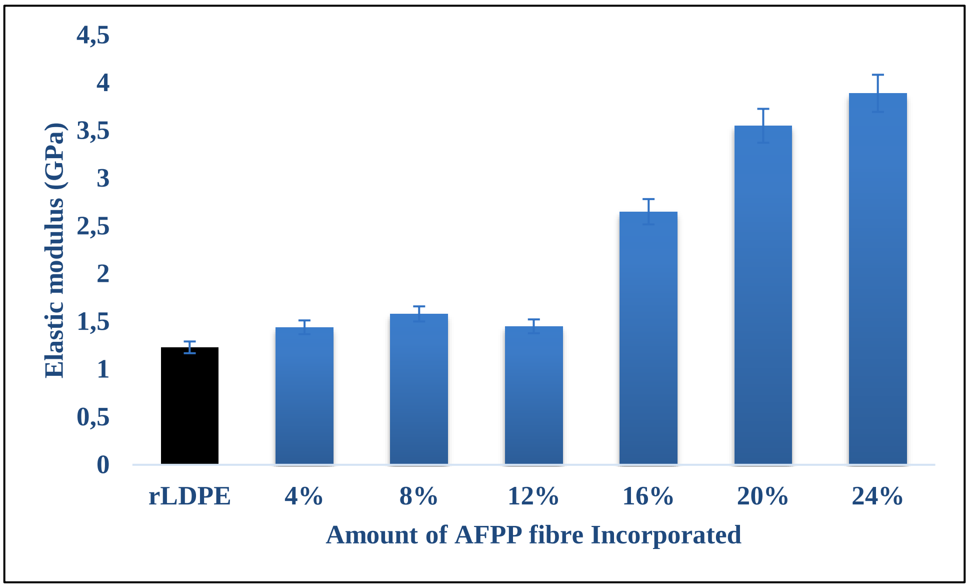


Figure 2: Effect of BA powder on the elastic modulus of recycled LDPE composites

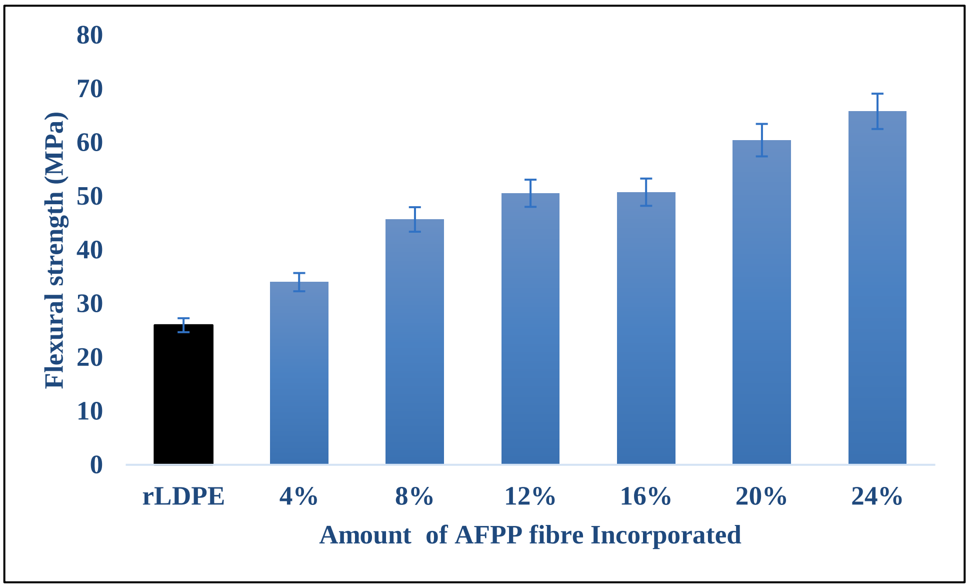


Figure 3: Effect of BA powder on the flexural strength of recycled LDPE composites

3.1.3 Effect of BA powder on the hardness value of recycled LDPE composites

Composite hardness testing is based on the surface's relative resistance to indentation by an indenter of specified dimensions under a specified load (Jacob *et al.*, 2018b). The hardness values of BAP reinforced waste LDPE composites were observed to increase with weight percentage of fibre from 4 % up to 24 %. The increase in hardness with weight percentage of fibre incorporated could be attributed to better interfacial adhesion between the fibres and the polymer matrix. Fibres act as load-bearing elements within the composite. When a load is applied, the fibres can transfer stress more efficiently than the matrix, leading to a higher resistance to indentation. Similarly, as the amount of BAP fibres incorporated increases, the distance between fibres decreases. This reduces the amount of matrix material that can deform under load, leading to higher hardness value. These results are at par with the work of Jacob (2019) and that reported by Jacob and Mamza (2020).

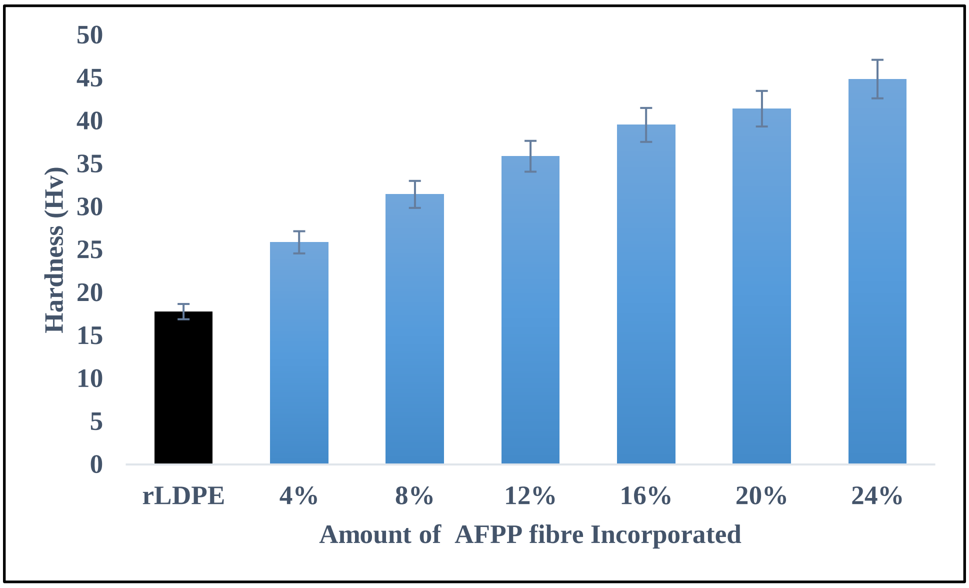


Figure 4: Effect of BA powder on the hardness value of recycled LDPE composites

3.2 Physical properties

3.2.1 Density

The density profile of the composites and unreinforced rLDPE is displayed in Figure 1. From the unreinforced (rLDPE) up to 24 % weight of BAP filled rLDPE composites, a progressive decrease in material density could be observed. The lowest was observed at 0.45 g/cm^3. This suggests that adding AFPP fibers to rLDPE reduces the material's density. These outcomes are comparable to those of other authors' works (Khalaf, 2015; Jacob, 2019; Jacob *et al.*, 2024).

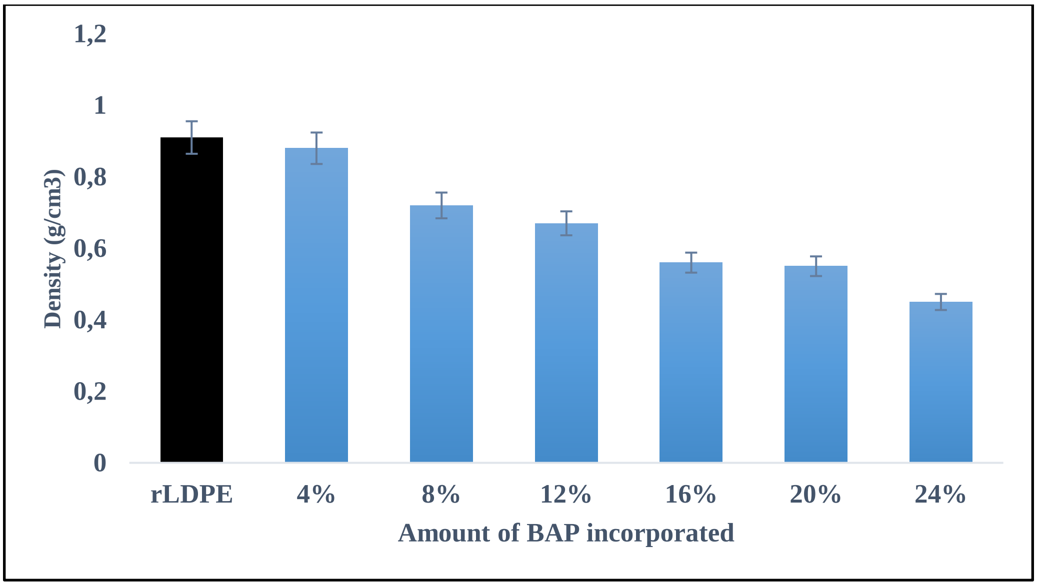


Figure 5: Effect of BAP fibres on the density of recycled LDPE composites

3.2.2 Water absorption

The water absorption rate of BAP-filled rLDPE composites is shown in Figure 6 following 240 hours of immersion at room temperature. Water absorption increased as the weight percentage of incorporated BAP fibre increased, reaching a maximum value of 6.65 % at 24 % of the weight fraction of fibre. This is expected, since moisture uptake in polymer composites increases with the amount of hydrophilic natural fiber incorporated. This outcome is consistent with research conducted by Abisha *et al*. (2023) and Jacob and Mamza (2020), who examined the mechanism of moisture absorption behaviour in waste HDPE composites filled with groundnut shell powder. Because rLDPE sample is hydrophobic, there is little to no moisture uptake, as shown by the linearity of the chart at 0 %.

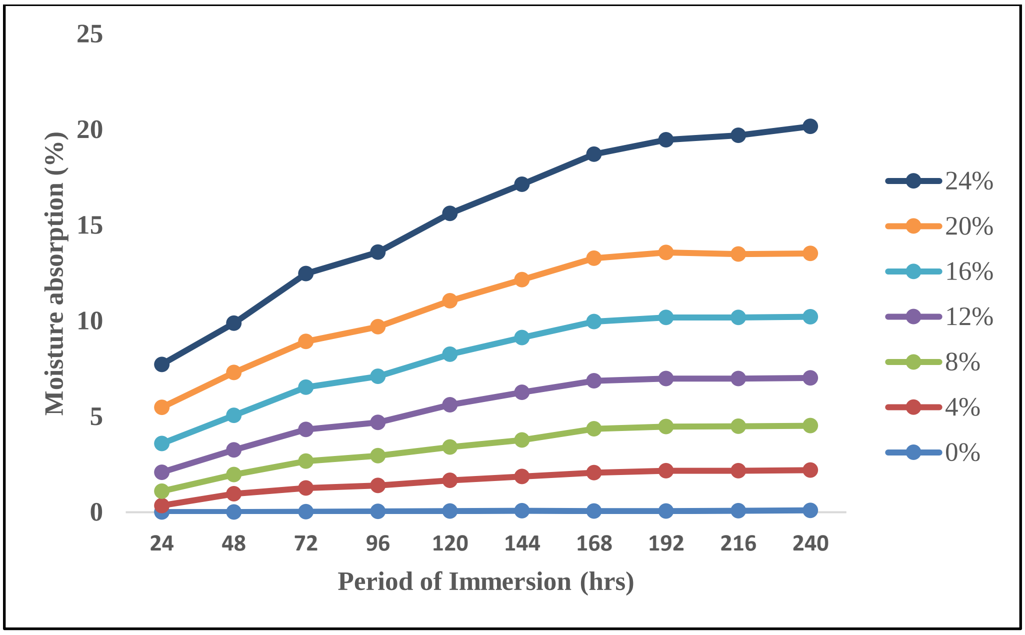


Figure 6: Moisture absorption behavior of BAP filled recycled LDPE composites

3.3 Thermal Properties

3.3.1 Dynamic mechanical analysis

3.3.1.1 Storage modulus of unreinforced recycled LDPE

The stiffness and energy storage capacity of a material are described by its storage modulus (Jacob *et al*., 2018a). In order words, it describes the energy stored in the system which depicts the elastic portion. Figure 7 shows the storage modulus of unfilled LDPE (control) at frequencies of 2, 5, and 10 Hz. The graph shows that the material is unstable at temperatures under 40 °C, with a maximum rigidity of 0.16 GPa. This finding is consistent with the work of Jacob *et al.* (2019b), who studied the thermomechanical properties of plantain peel particle reinforced waste LDPE as composite wall tiles and that reported by Jacob (2023) who also reported the effect of variation in frequency on the dynamic mechanical properties of plantain peel particulate reinforced recycled polypropylene composites. The results also indicates that incorporation of plantain peel particulate improved the thermal stability of recycled polypropylene.

3.3.1.2 Storage modulus of BAP reinforced recycled LDPE composites at different compositions.

Figures 8-13 show the storage modulus of 4 %, 8 %, 12 %, 16 %, 20 % and 24 % BAP-$BaSO_4$ reinforced rLDPE composites at 2, 5, and 10 Hz, respectively. These results show that the addition of the fibre into recycled LDPE increased the material's stiffness and energy storage capability. This is because LDPE's maximum stiffness stability increased from 0.16 GPa (unreinforced rLDPE; Figure 7) to: 0.38 GPa at 4 % weight of BAP (Figure 8), 0.42 GPa at 8 % BAP (Figure 9), 0.58 GPa at 12 % BAP (Figure 10); 0.94 GPa at 16 % BAP (Figure 11); 1.20 GPa at 20 % GPa (Figure 12); and 1.60 GPa at 24 % (Figure 13) weight percentage of the BAP incorporated. The obvious evidence of an increase in storage modulus from 4 % to 20 % weight percentage of (BAP) fibres could be attributed to the composites' strong fiber-matrix interaction (Jacob *et al.*, 2019b). As the weight fraction of reinforcement increases, the interfacial area also increases, potentially leading to improve adhesion and enhanced load transfer which in turn increases the composites stiffness stability. It should also be noted that the reinforcement can hinder the molecular motion of the matrix polymer chains, limiting their ability to deform under stress. This restriction of deformation contributes to an increase in the storage modulus (Gupta, 2018; Jacob, 2023; Jacob *et al.*, 2023; Jacob *et al.*, 2024).

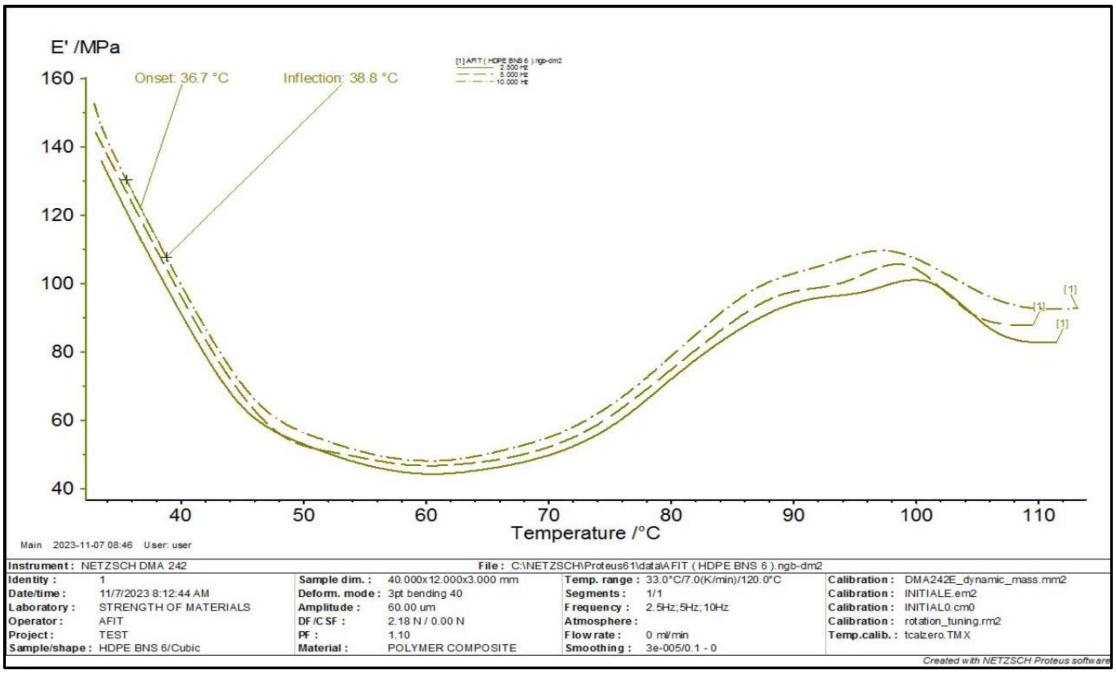


Figure 7: Storage modulus of unreinforced recycled LDPE at 2, 5 and 10 Hz

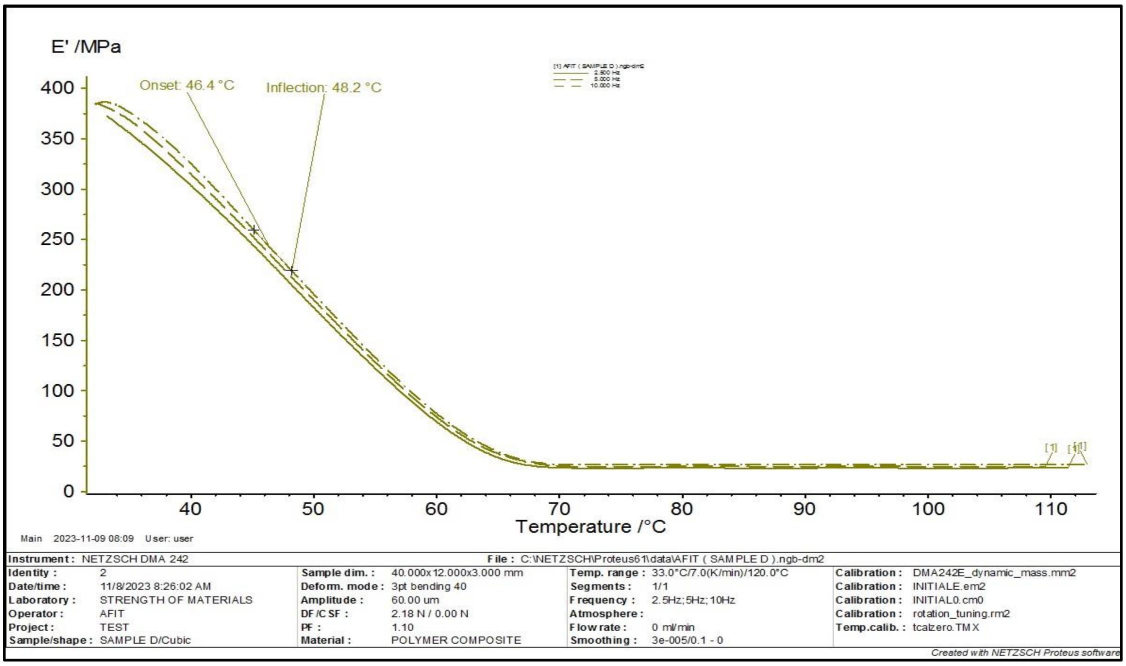


Figure 8: Storage modulus of 4 % BAP-$BaSO_4$ filled rLDPE composites at 2, 5 and 10 Hz

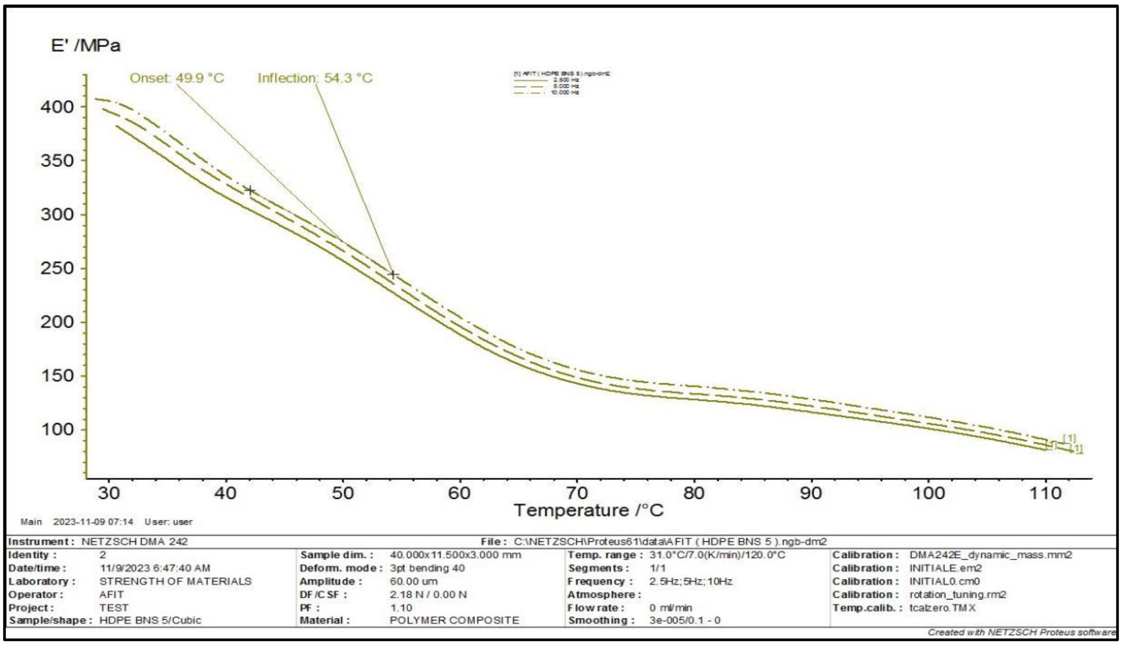


Figure 9: Storage modulus of 8 % BAP-$BaSO_4$ filled rLDPE composites at 2, 5 and 10 Hz

The curves also show that the composites are stable under dynamic loading with increasing temperature up to 36.8 ℃ before its point of inflection at 48.2 ℃ (Figure 7); 54.3 ℃ (Figure 8); 47.3 ℃ (Figure 9), 76.8 ℃ (Figure 10); 57.6 ℃ (Figure 11); up to 76.1 ℃ (Figure 12) which are taken as the glass transition temperatures for the composites with 4, 8, 12, 16, 20 and 24 % weight of BAP fibre, respectively. Glass transition temperature marks the temperature at which a material transitions from a hard, brittle state (glassy state) to a soft, flexible state (rubbery state). It is a crucial property of amorphous materials, particularly polymers. According to Palanivel *et al.* (2017), Jacob *et al.* (2019b) and Jacob and Yusuf (2023), these materials are suitable for applications up to maximum temperature of 70 ℃ at 24 % weight of BAP incorporated. The dynamic mechanical analysis results (storage modulus) are in agreement with the determined mechanical properties of acetylated BAP filled recycled LDPE composites.

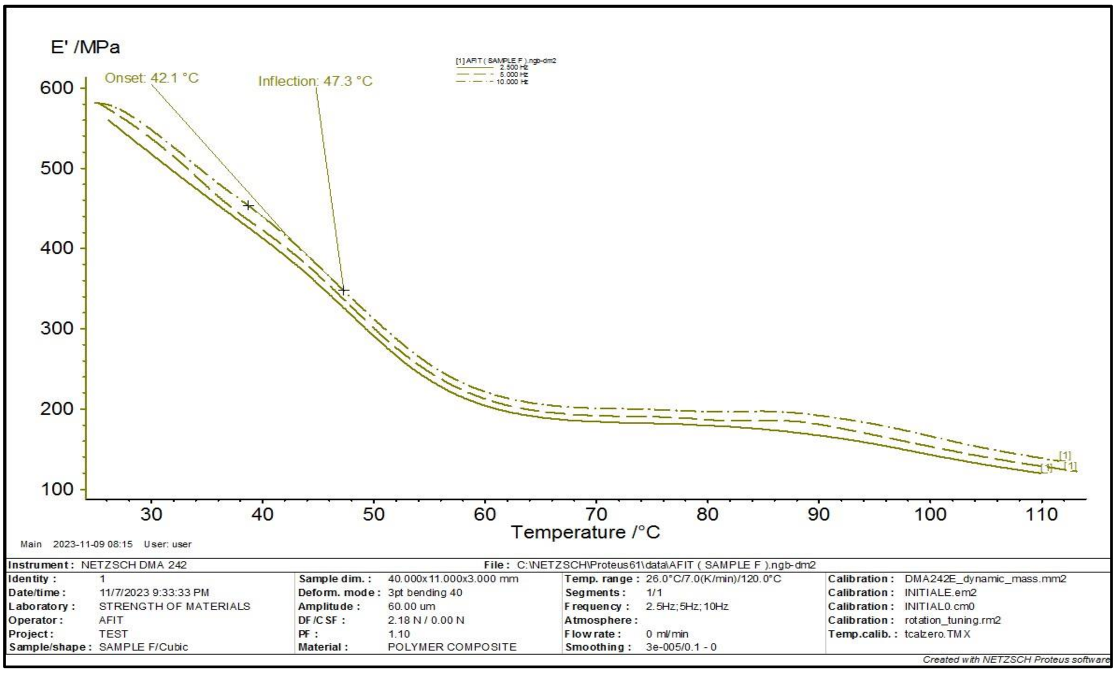


Figure 10: Storage modulus of 12 % BAP-BaSO4 filled rLDPE composites at 2, 5 and 10 Hz

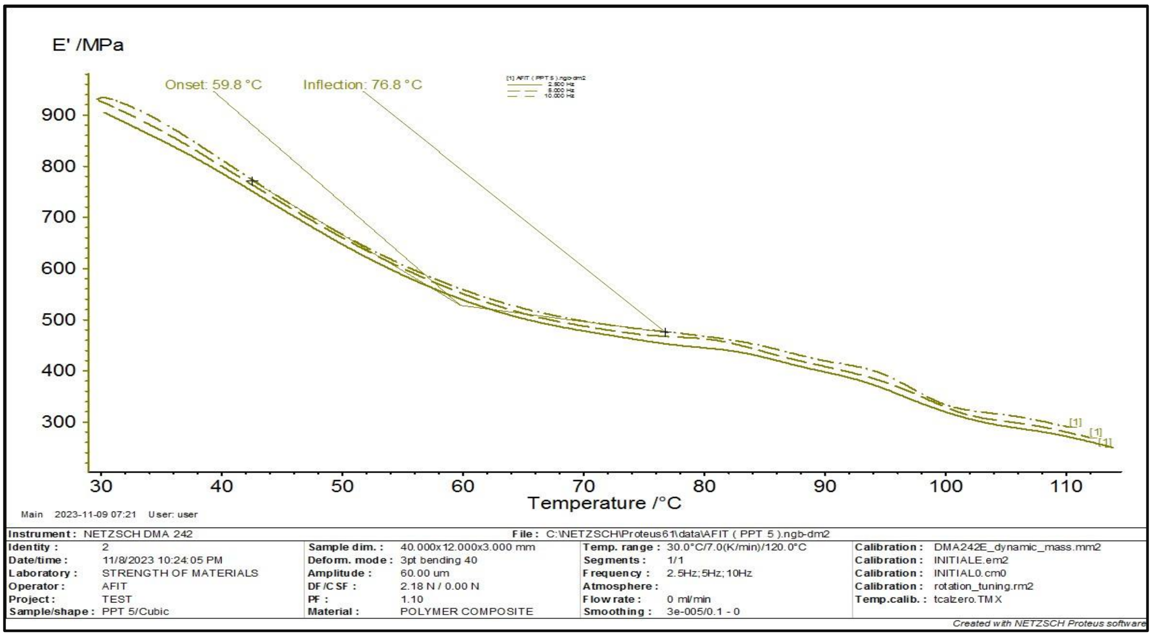


Figure 11: Storage modulus of 16 % AFPP-$BaSO_4$ filled rLDPE composites at 2, 5 and 10 Hz

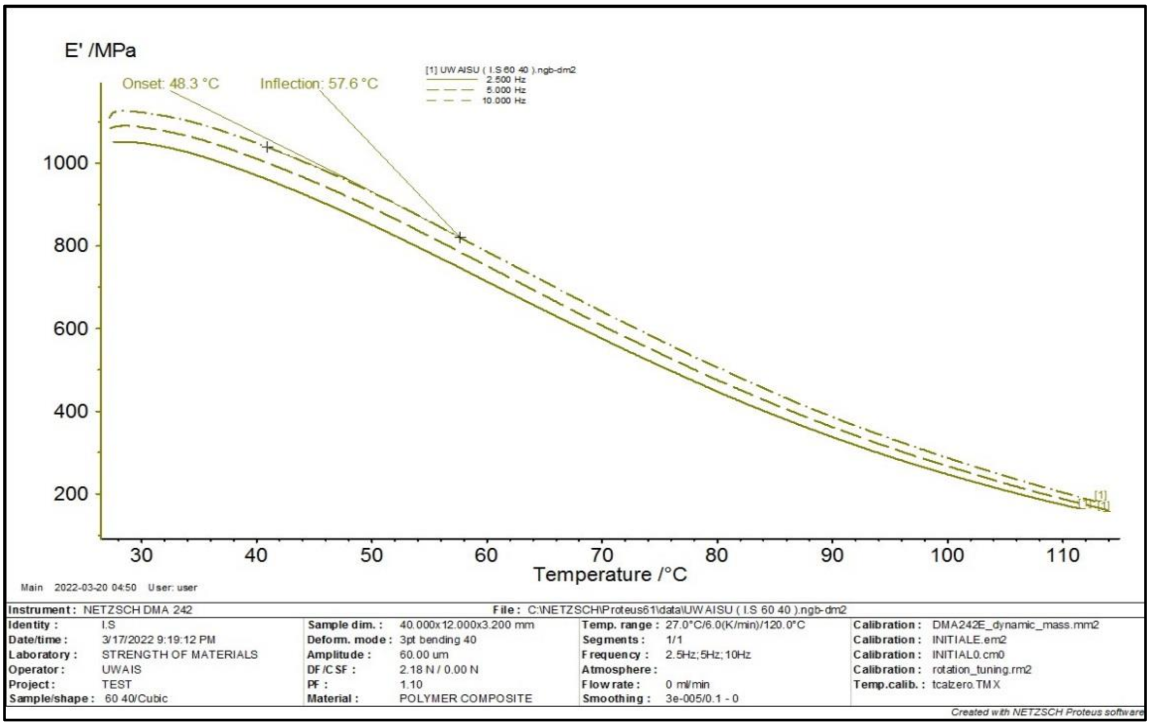


Figure 12: Storage modulus of 20 % AFPP-$BaSO_4$ filled rLDPE composites at 2, 5 and 10 Hz

3.3.1.3 Damping parameter of BAP reinforced waste LDPE composites at different compositions.

Damping (Tan delta) is defined as the loss modulus divided by storage modulus. It is also known as the loss factor and is typically stated as a dimensionless number (Gupta, 2018; Jacob *et al.*, 2019a; Rajesh *et al.*, 2021). A high value of damping is indicative of a material with high non-elastic strain behaviour while low value of damping indicates that the material is more elastic (Jacob *et al.*, 2019b). Figure 14 depicts the damping parameter of unfilled rLDPE, whereas Figures 15-17 depict the damping parameters of BAP-filled composites at 15, 20, and 25 %, respectively. These results show that the damping of the composites reduces with the weight fraction of reinforcement from 4 % to 24 %. This suggests that the sample with 24 % BAP has the best load bearing capability of all the composites. This could be due to improved interfacial interaction between the rLDPE and AFPP fibres. Other authors have shown similar decrease in damping with weight percentage of reinforcement (Gupta, 2018; and Jacob *et al*., 2019a). The figures also show the glass transition temperature (the temperature at which an amorphous solid material changes from a hard, brittle state to a softer, more pliable state) of unfilled rLDPE and the composites. Figure 14 shows that the unfilled rLDPE sample has a glass transition temperature of 50.7 ℃, which increases to 61.4 ℃ with 15 % BAP (Figure 15), 77.1 ℃ with 20 % weight percentage of fibre, and drops to 68.6 ℃ with 25 % weight percentage of fibre. This suggests that incorporating AFPP fibre into rLDPE increases stiffness stability (Jacob, 2023; Jacob and Yusuf, 2023, Jacob *et al*., 2023).

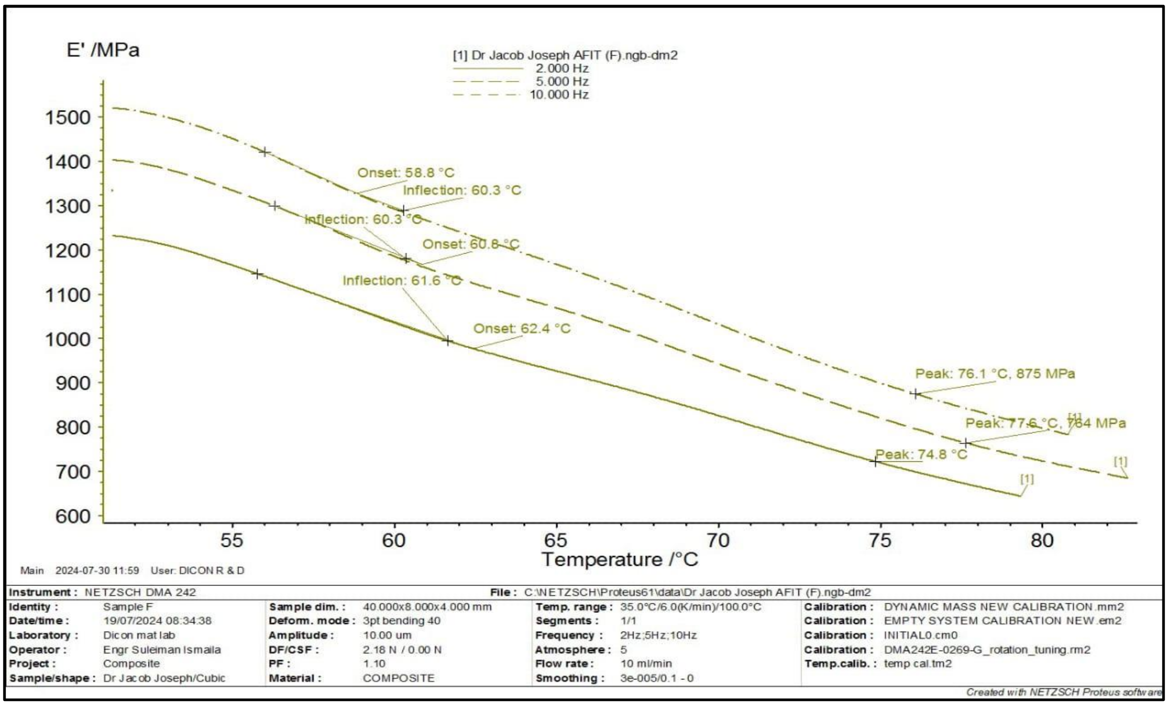


Figure 13: Storage modulus of 24 % BAP-$BaSO_4$ filled rLDPE composites at 2, 5 and 10 Hz

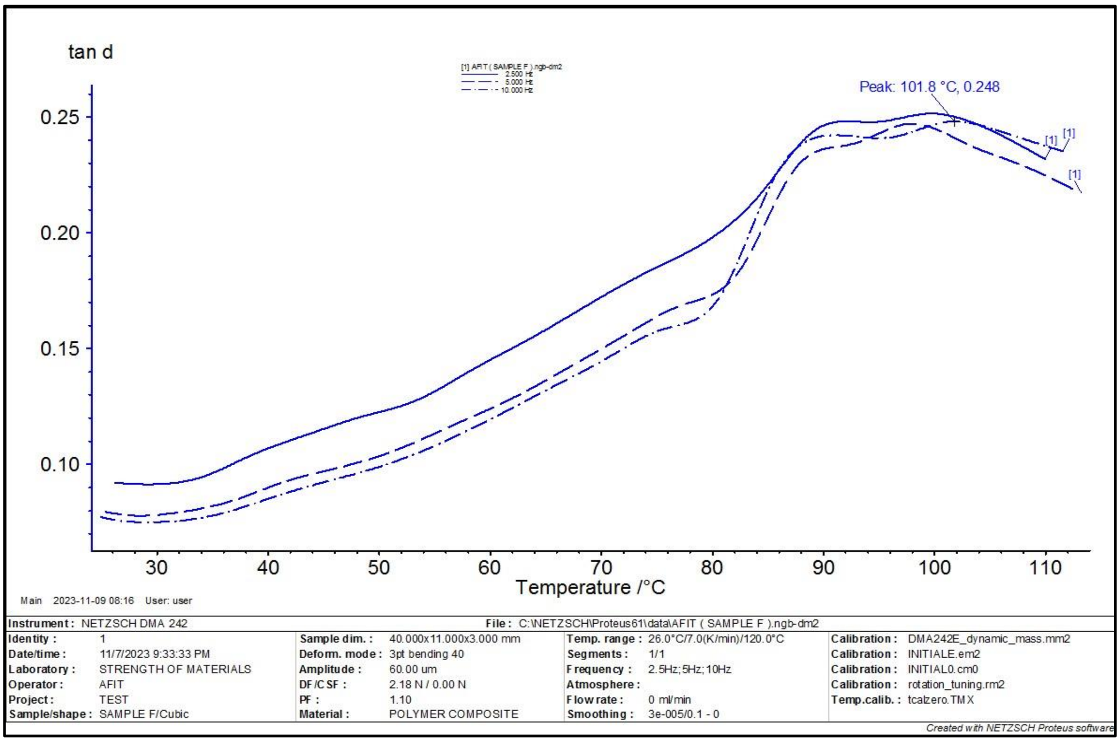


Figure 14: Damping parameter of unreinforced waste LDPE at 2, 5 and 10 Hz.

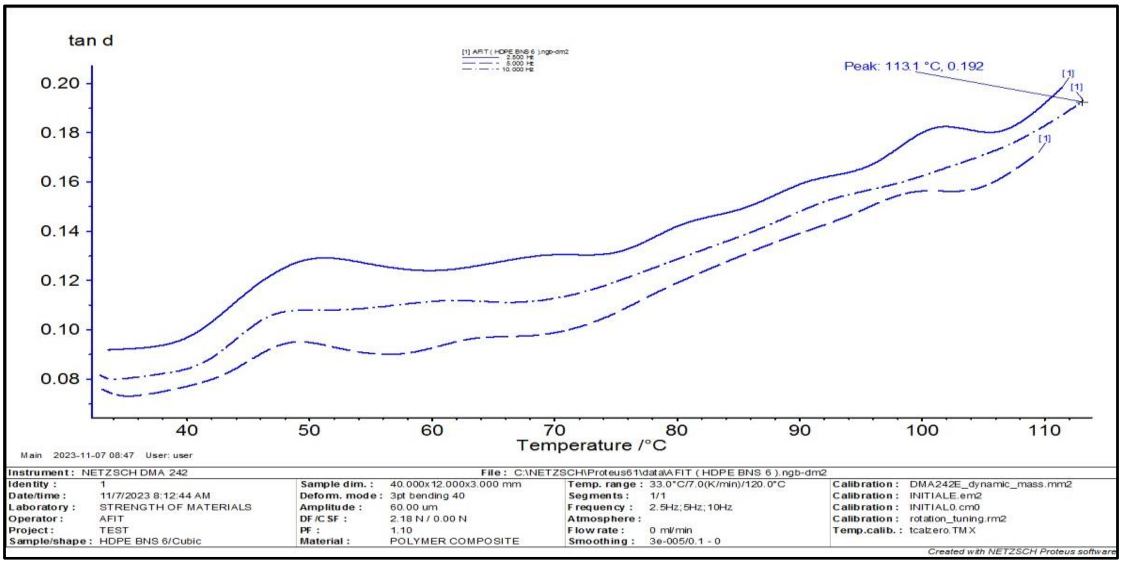


Figure 15: Damping parameter of 4 % BAP-$BaSO_4$ reinforced waste LDPE composites at 2, 5 and 10Hz

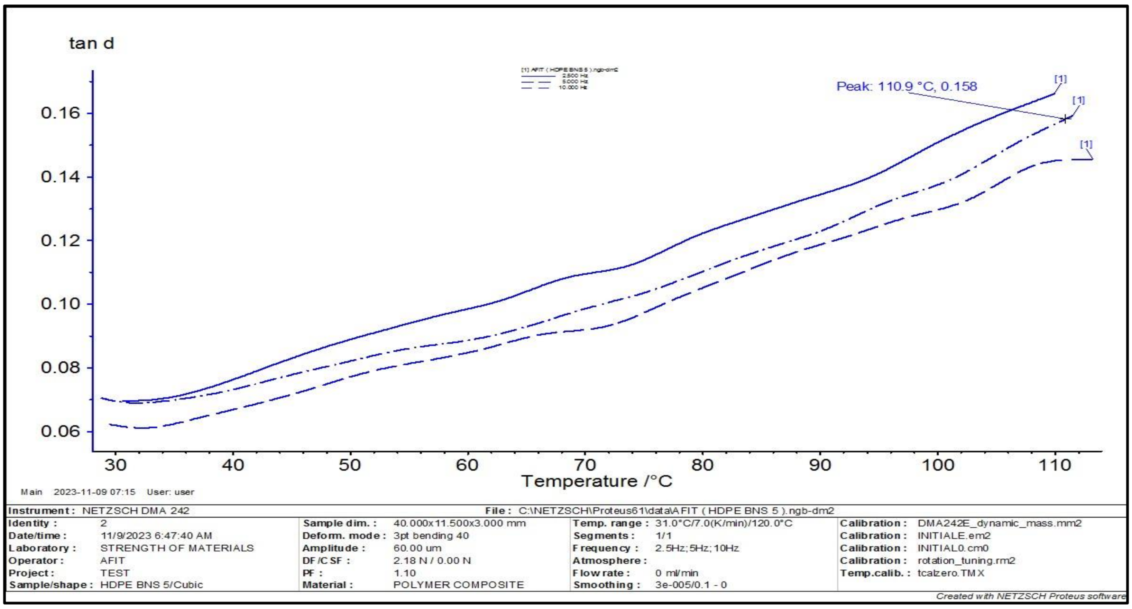


Figure 16: Damping parameter of 8 % BAP-$BaSO_4$ reinforced waste LDPE composites at 2, 5 and 10 Hz

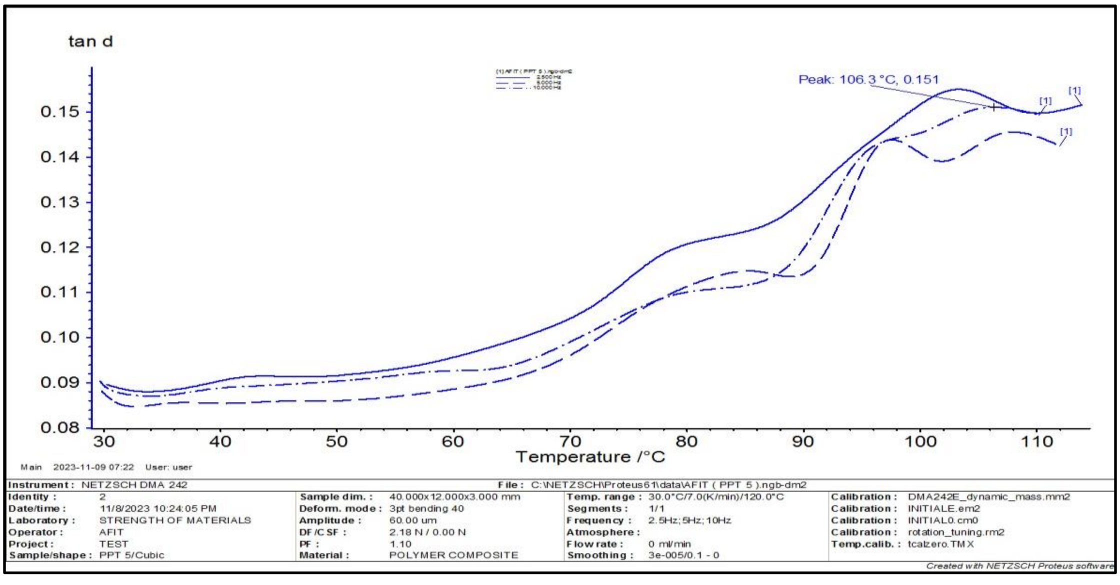


Figure 17: Damping parameter of 12 % BAP-$BaSO_4$ reinforced waste LDPE composites at 2, 5 and 10 Hz.

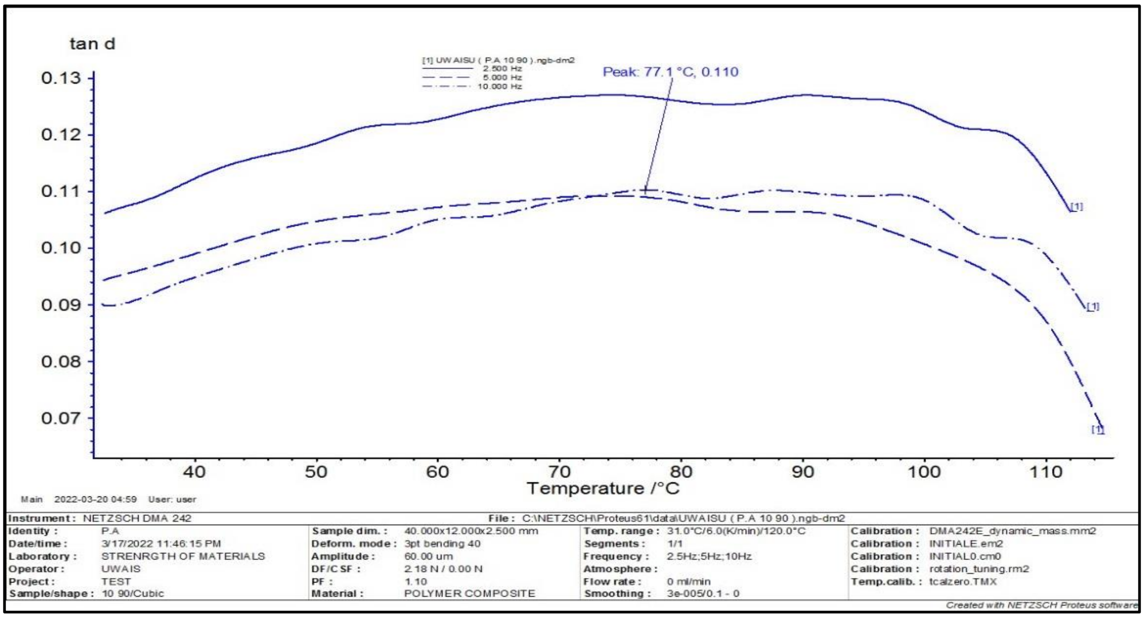


Figure 18: Damping parameter of 16 % BAP-$BaSO_4$ reinforced waste LDPE composites at 2, 5 and 10 Hz

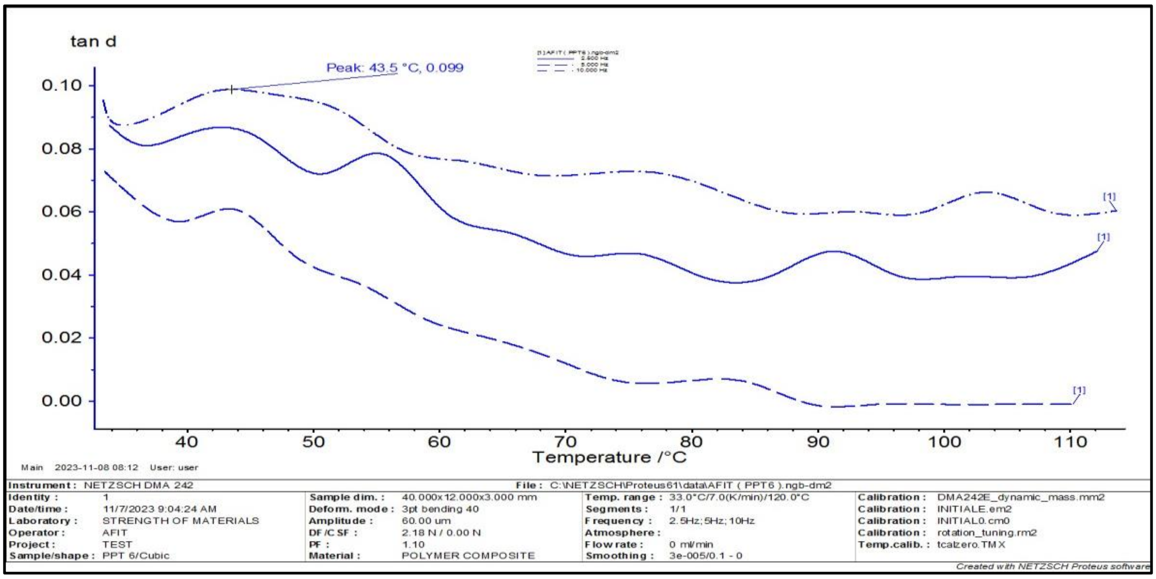


Figure 19: Damping parameter of 20 % BAP-$BaSO_4$ reinforced waste LDPE composites at 2, 5 and 10 Hz.

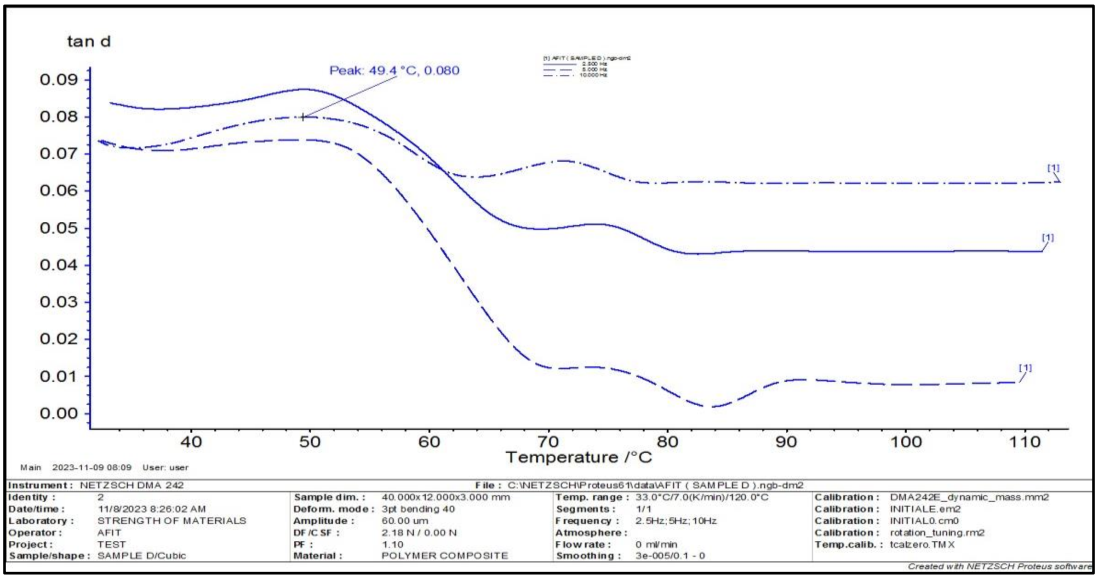
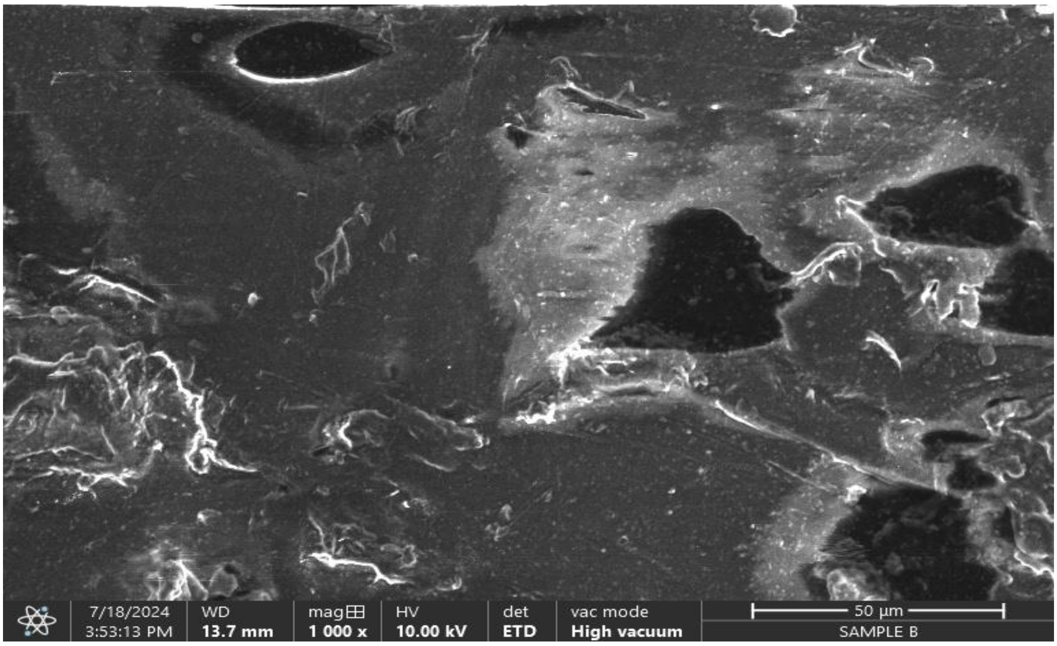


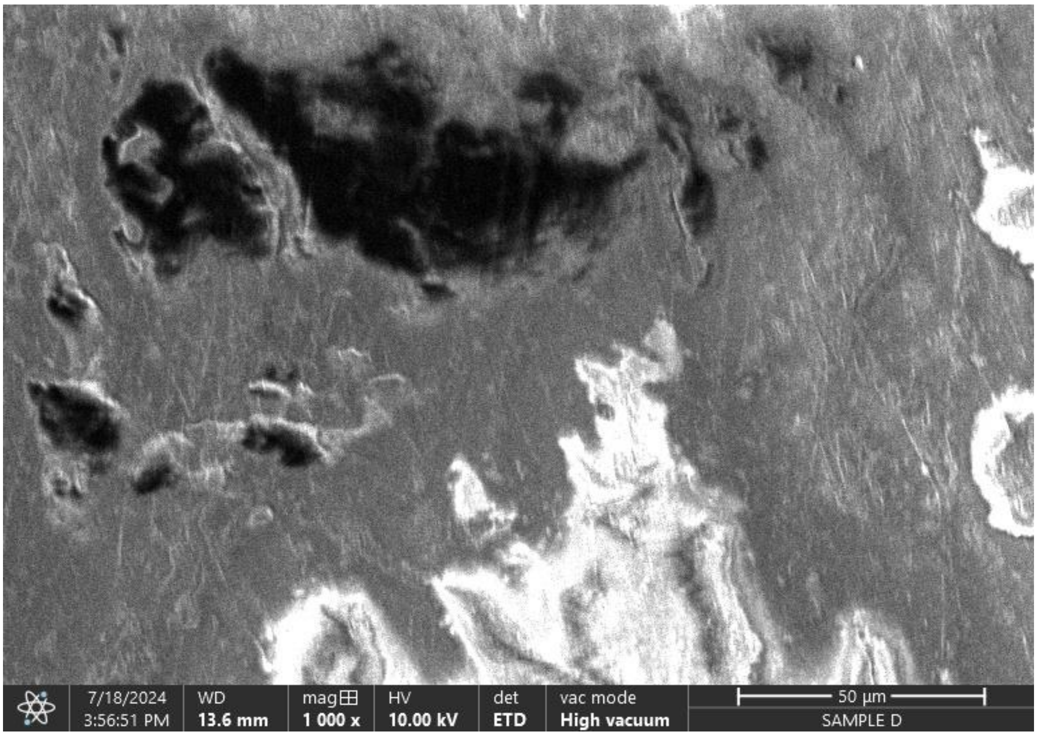
Figure 20: Damping parameter of 24 % BAP-$BaSO_4$ reinforced waste LDPE composites at 2, 5 and 10 Hz.

3.4 Morphological Properties of BAP filled recycled LDPE Composites

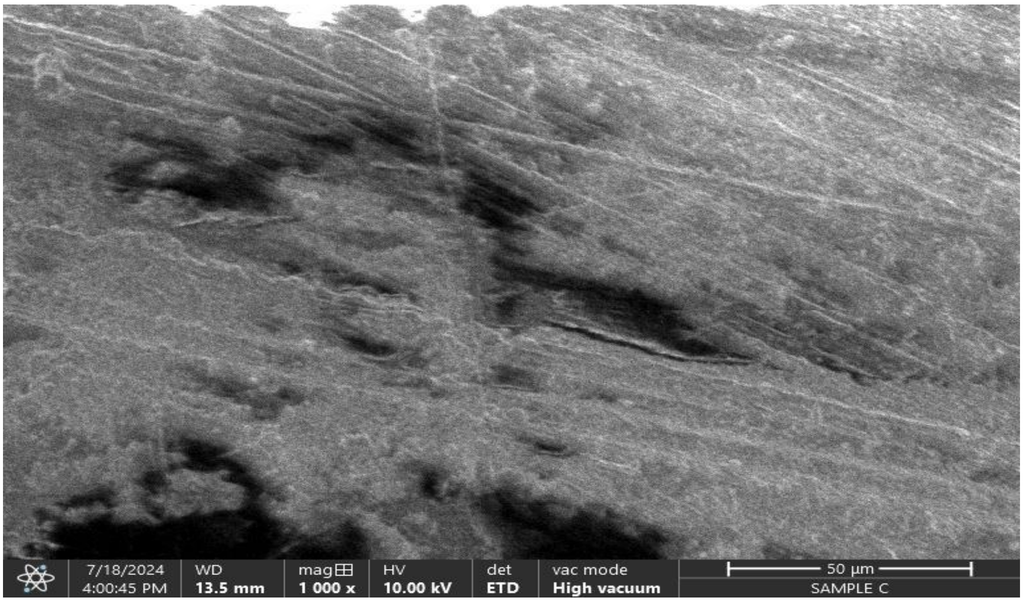
SEM micrographs (Plates I–IV) show the morphological properties of BAP-recycled low-density polyethylene composites. The presence of voids was detected in the unreinforced rLDPE (plate I) and was found to reduce with the addition of 16 % BAP (plate II). Better dispersion of BAP fibres in rLDPE could be observed as the amount of BAP fibres incorporated increased (plates III and IV). As a result, the SEM data show that the treated BAP fibres interact more effectively with the rLDPE matrix. This demonstrates that the increased physical, mechanical, and dynamic mechanical properties of BAP-rLDPE composites were due to improved interfacial adhesion between the fibres and the rLDPE matrix



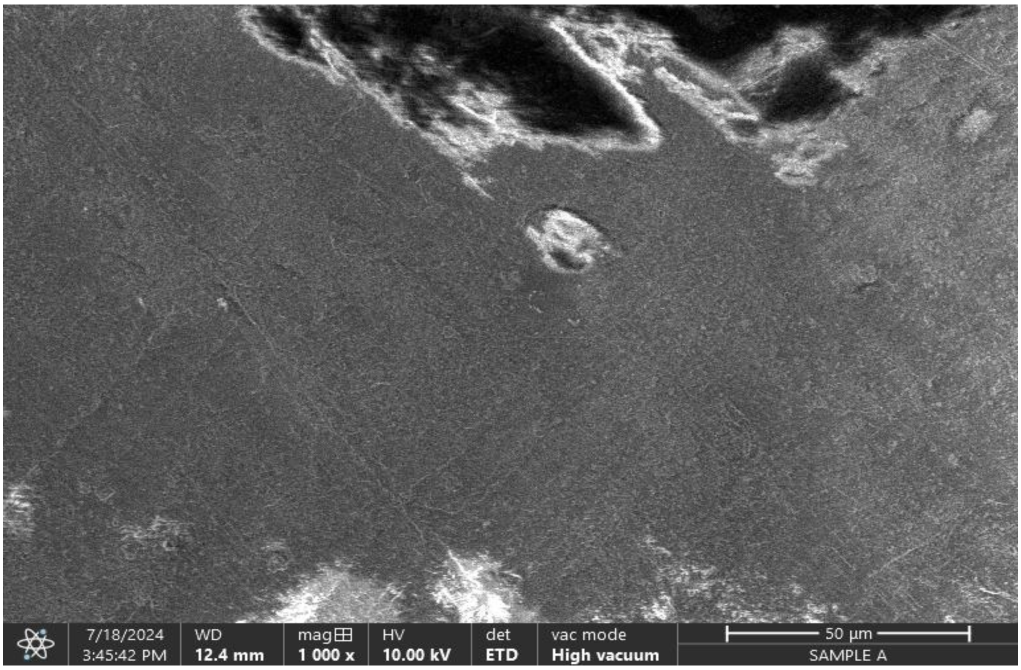
(I)



(II)



(III)



(IV)

Plate I-IV: SEM micrographs with (I) recycled LDPE at x 1000; (II) 16 % BAP at x 1000; (III) 20 % BAP at X 1000 and (IV) 24 % BAP at accelerating voltage of 10 kV.

Conclusion

The effects of AFPP powder addition on the physical, mechanical and dynamic mechanical characteristics of recycled LDPE composites were studied. The results obtained lead to the following conclusions:

The physical, mechanical, and visco-elastic qualities of recycled LDPE were enhanced by the addition of African fan palm powder. In comparison to 0.91 g/cm^3 of unreinforced rLDPE, the density dropped with the weight percentage of AFPP fibres, reaching a minimum of 0.45 g/cm^3. Water absorption was only 6.65 % in the 24 % weight fraction. With a 24 % weight percent of reinforcement, the material's ideal tensile strength of 55.5 MPa, stiffness of 3.89 GPa, flexural strength of 65.8 MPa, and hardness value of 45 Shores were attained. Dynamic mechanical properties showed that a composite with 24 % reinforcement also had the highest storage modulus and load bearing capability (1.60 GPa) at 24 % weight of BAP fibre compared to 160 MPa of unreinforced.

The results of physical, mechanical and thermal properties of BAP filled recycled LDPE composites indicate that these properties of recycled LDPE have been enhanced with the incorporation of *Borassus aethiopum* powder and $BaSO_4$ as nucleating agent. Scanning electron micrographs results further affirms the observed improved properties of the composites at higher weight fraction of reinforcement.

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