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Original Research Article

Mechanical and Morphological Properties of Fluted Pumpkin Stem Fiber (Telfaira occidentalis) Recycled High Density Polyethylene Nanocomposites

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Abstract: The outstanding characteristic properties of nanographene such as high aspect ratios and improved mechanical properties in comparison with other types of nanofillers like carbon nanotubes and clays has accelerated their use as nanosize filler in polymer matrix composites. The litter of agro-wastes is a critical issue which must be addressed for the preservation of the global environment. The effect of nanographene addition on the mechanical and morphological properties of nanocomposites prepared using fluted pumpkin stem fiber (FPF) and recycled high density polyethylene (R-HDPE) was investigated. Four weight levels of nanographene 0, 0.5, 1.5 and 2.5 wt % were mixed with 65 wt. % HDPE, 35wt. % FPF and 3wt% maleic acid anhydride produced using melt compounding and the extruded nanocomposites was shaped by injection molding machine for the mechanical tests. The results showed that addition of 0.5 wt % nanographene increased the flexural strength, flexural modulus and notched impact strength by 24.77MPa, 1800MPa and 32.61J/m^2, compared to the control samples 20.5MPa, 1500MPa and 19.35J/m^2 respectively. Although the addition of nanographene to the FPF/HDPE composite significantly improved the flexural strength, flexural modulus and notched impact strength, these improvements came at a unique nanographene loading of 0.5 wt %. The morphological images revealed that the samples with nanographene loading of 0.5 wt. % showed no fiber pullout/holes, whereas higher contents (1.5-2.5wt %) of nanographene exhibited fiber pullout/holes and were easily agglomerated. This study has shown that fluted pumpkin stem fiber could be used in composite formulation with good results comparable to wood-plastic-composites.

Keywords: Nanocomposites, polyethylene, agro-waste, mechanical properties, morphology.

INTRODUCTION

Nanotechnology has broadened the verified versatility of plastic-based materials in emerging technologies. The surge in the utilization of plastics has contributed to the ballooning global solid waste problems, of which plastic materials account notably for the upsurge and are predominantly made of polyolefins such as polyethylene and polypropylene. Particularly in growing economies, incineration, uncontrolled dumping, and land filling are primary routes of disposal of these wastes. Thus, environment-friendly and energetically cheap methods of exploiting this huge amount of trash have become a worldwide problem. Therefore, legislators around the world having come to awareness that indiscriminate dumping and incineration are not environment-friendly ways of disposing these waste has called for stoppage, and at the same time campaigning for recycling and reuse of these polymer based solid wastes as remedy to this confrontations [1].

As a result of rising environmental and ecological apprehension, composites made of thermoplastics filled/reinforced with cheap lignocellulosic fibers have enkindled tremendous interests due to earmarked advantages such

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as reduced wearing of processing equipment and low temperatures of processing in comparison with the routinely accessible mineral fillers by that performing a phenomenal part in economizing energy in the course of composites fabrication [2].

Wood-plastic composite (WPC) comprises of polymer and lignocellulosic material combined together in different proportions [3]. WPCs can be generally considered to be eco-benign. Wood plastic composites have a woody appearance but they are much more efficient with respect to solid wood. The useful features of wood-plastic-composites include lower moisture absorption capacities, oxidation resistance, diffusion resistance and resistance to wreck by insects, lesser densities, more durable, greater dimensional stability, good physical and mechanical properties, facileness, and ability to accept decorative paintings. The use of wood and non-wood plant fiber as reinforcements in thermoplastics has increased dramatically in recent years [4].

There is no gainsaying the fact that by using nanomaterials the properties of composites currently attained in the industrial sector can be upgraded, thereby facilitating the production of new products with improved value and greater efficiency [5].

A considerable amount of plastic wastes enter daily into the environment and its recycling is good for the economy and the environment. Generally, the constituents of plastics in municipal solid waste is essentially the commodity (widely used thermoplastics) thermoplastics which includes polyethylene (PE), polypropylene (PP), polystyrene (PS), polyvinylchloride (PVC) and polyethylene terephthalate (PET) [6].

Recently, approaches based on nanotechnology are being considered to reinforce composites. Graphene is notable among the different types of nanosize fillers that are used in the manufacture of wood-plastic-composites in recent years because of its exceptional properties: mechanical, electrical, heat and optical [7-9].

Graphene is a two-dimensional compound of carbon formed by the sp^2 orbitals of the carbon atom [10-13]. The intrinsic properties of graphene allow even small additions to significantly improve the properties of composites [8]. Graphene's superior properties compared with the matrix polymers in graphene/polymer nanocomposites results in high mechanical, heat resistance, gas permeability, electrical and flammability resistance compared with pure polymer [14-16]. There is superior enhancement in mechanical properties of polymer composites on the addition of graphene compared to clay or other nanofiller nanocomposites [17, 18].

Literature review has shown that wood-plastic composites made of poplar wood flour/ polypropylene polymer matrix and nanographene sheets showed improved bonding and adhesion between particles and are enhanced by maleic anhydride grafted with polypropylene [9]. These experiments showed that when the amount of graphene of 0.8% was used, the flexural and tensile properties reached their peak. This was caused by the lack of proper stress transfer and agglomeration when using graphene in the amount of 3 to 5 wt %.

The effect of graphene addition on the physical and mechanical properties of polypropylene/bagasse fiber composites was investigated by Chacharmahali [19]. It was reported that increase in strength was obtained in the samples made with 0.1 % graphene, an increase of fiber from 15 to 30% increased the tensile and flexural strengths, whereas, the impact strength was decreased.

The mechanical properties of linear low density polyethylene (LLDPE) reinforced with dodecyl amine-modified graphene was investigated by Kuila [20]. Their study showed improvements in the storage moduli and thermal stability of the nanocomposites with increasing dodecyl amine-modified graphene content.

Chatterjee *et al*., [21] examined the tensile and crystallization properties of fiber polyamide 12 reinforced with graphene and carbonic nanotubes. The crystallization index of polyamide fiber increased after adding nanofillers due to their nucleating effect. Also, flexural and tensile strengths as well as modulus were increased by adding the nanofillers. The increase in modulus was very significant and meaningful.

This research investigates the effect of nanographene loadings on the mechanical and morphological properties of fluted pumpkin stem (Telfaira occidentalis)/recycled high density polyethylene nanocomposites.

MATERIALS AND METHODS

Materials

Preparation of fluted pumpkin stems flour

The fluted pumpkin stems were collected as agricultural waste raw material from local sources in Awka metropolis, Anambra State, Nigeria. The collected fluted pumpkin stems were washed with water to remove any dirt and then cut into small pieces. Next, the fluted pumpkin stems were dried in oven at 70°C until a constant weight of about 2% was obtained. The dried fluted pumpkin stems were further ground into powder using a grinder as shown in Plates 1, 2 and 3 below. The fluted pumpkin flour was sieved using a 100 mesh stainless steel.

Plate 1: Fluted Pumpkin Stems Agro-waste

Plate 2: Edible Fluted Pumpkin Leaves

Plate 3: Grinded and oven dried fluted pumpkin stem flour

Collection of Plastic

The polyethylene plastic wastes were collected from MCC dumpsite in Awka, Anambra State, Nigeria and using physical identification of plastics polyethylene waste were sorted out.

Plastic Preparation

The polyethylene plastic waste materials (HDPE) were first taken to the hydraulic cutting machine to cut the plastic lumps into smaller sizes before transferring to the grinding/crushing machine for grinding/crushing and scaling. The ground/crushed plastics waste materials were poured into a rectangular washing bowl filled with clean water and the polyethylene (HDPE) that floated were collected by the use of a sieve and the ones that sank were discarded. The washed plastics waste materials were sun dried for 24 h and packaged for further use, as shown in Plate 4.

Plate 4: Recycled/crushed polyethylene (HDPE)

Maleic acid anhydride grafted polyethylene (PE-g-MA)

To improve compatibility between the fluted pumpkin stem flour and recycled high density polyethylene, 3wt % of polyethylene grafted anhydride (PE-g-MA) as compatibilzer used for better dispersion of nanographene in the polymer matrix.

Nanographene

Type AO4 graphene nanoparticles were purchased from Graphene Supermarket Company USA. The characteristic property of the nanographene used is displayed in Table 1 below.

Table 1: Characteristics property of Nanographene

Specific surface (m^2g^{-1})	Color	Purity (%)	Average thickness (mm)	Length of particles (µm)
More than 15	black	98.5	60	3-7

METHODS

Mixing of Material

The recycled/crushed HDPE, fluted pumpkin flour, PE-g-MA, and nanographene were mixed together as shown in Table 2. The materials were mixed in a HAAKE SYS 9000 internal mixer (De Soto, MO USA) at the Materials Engineering Workshop, Nnamdi Azikiwe University, Awka, Nigeria for 8 min at a mixing temperature of 160^{o}C and stirring speed of 60 rpm to achieve constant torque. After mixing, the components were bagged according to sample number as shown in Table 2, prior to subsequent processing.

Preparation of Composites

Table 2 shows the formulation of the mixes and the symbols used for the different mixes prepared. The various mixes of the fluted pumpkin stem flour, recycled polyethylene, nanographene and MAPE were poured into the hopper feeder of a co-rotating twin-screw extruder. The temperature of the extruder barrel was maintained at a temperature of 160^{o}C at a screw speed of 50 rpm and melt pressure of 10 psi. The extrudate that emerged from the extruder nozzle were initially allowed to cool by immersing in a water bath and were later grounded. The granules were injection molded at a temperature of 160^{o}C using an injection molding machine to produce samples according to ASTM standard. To attain equilibrium moisture content prior to mechanical tests samples were stored for two weeks in a conditioning room at 23±2°C and 50±5% relative humidity.

Table 2: Composition of HDPE/Fluted Pumpkin Stem Flour

Sample No	FPF (wt %)	R-HDPE (wt %)	NG (wt %)	PE-g-MA (wt %)
S1	35	62	0	3
S2	35	61.5	0.5	3
S3	35	60.5	1.5	3
S4	35	59.5	2.5	3
FPF= fluted pumpkin flour; rHDPE=recycled high-density polyethylene; NG=nanographene; MAPE=maleic acid grafted polyethylene				

Mechanical Properties

Flexural testing

Flexural properties were measured for flexural strength and flexural modulus tests according to ASTM D 790 10 (ASTM International 2010). Five samples of 203 x 38 x 10 mm were cut and stored for 48 h at 23±2°C and 50±5% RH in a conditioning room. The test span was 152 mm, and an Instron Universal Testing Machine (Model 4466) was used for the test. The machine was equipped with 8.5kN load cell with a support span of 20 times the depth of the beam with a crosshead speed of 5mm min^{-1}.

Notched Izod Impact testing

Notched impact strength properties were conducted according to ASTM D 256 10 (ASTM International 2010). The tests were carried out using a pendulum machine. Five samples of 63 x 12 x 3 mm were cut and stored for 48 h at 23±2°C and 50±5% RH in a conditioning room. The pendulum was calibrated by running the striker at the zero position to compensate for any frictional error.

Morphological properties

The morphological properties of the composites were analyzed using a scanning electron microscope (SEM). SEM images (micrographs) of the fracture surfaces of the specimens after flexural test were obtained using SEM (S-570 Quanta). Prior to the analysis the specimens were sputter coated with a thin film of gold in order to avoid electrical charges build-up. A 7kV accelerating voltage was applied during the examination.

RESULTS AND DISCUSSIONS

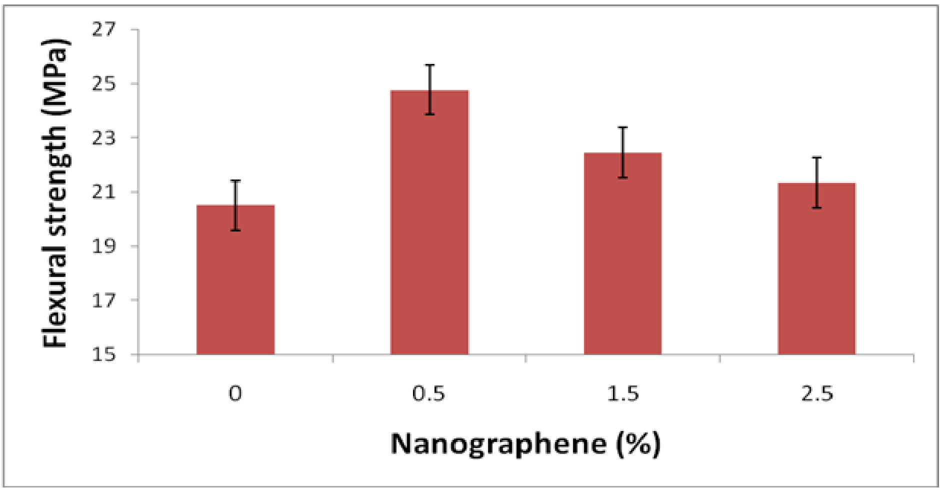

Fig 1: Nanographene loading vs. flexural strength of FPF/HDPE nanocomposite

The effect of nanographene content on the flexural strength of FPF/HDPE composites is shown in Figure 1. The results showed that the maximum (24.77MPa) and minimum values (20.5 MPa) average flexural strength were obtained at 0.5% and 0% wt of nanographene loading, respectively. The flexural strength of FPF/HDPE composites increased up to 0.5 wt % of nanographene and then decreased with additional nanographene loading. The mechanical strength of some composites can be increased by adding nanoparticles [16]. Increased mechanical properties as a result of adding low levels of nanoparticles such as nanographene, is easily understood because nanoparticles transfer stress and improve the connectivity of the composite components [27].

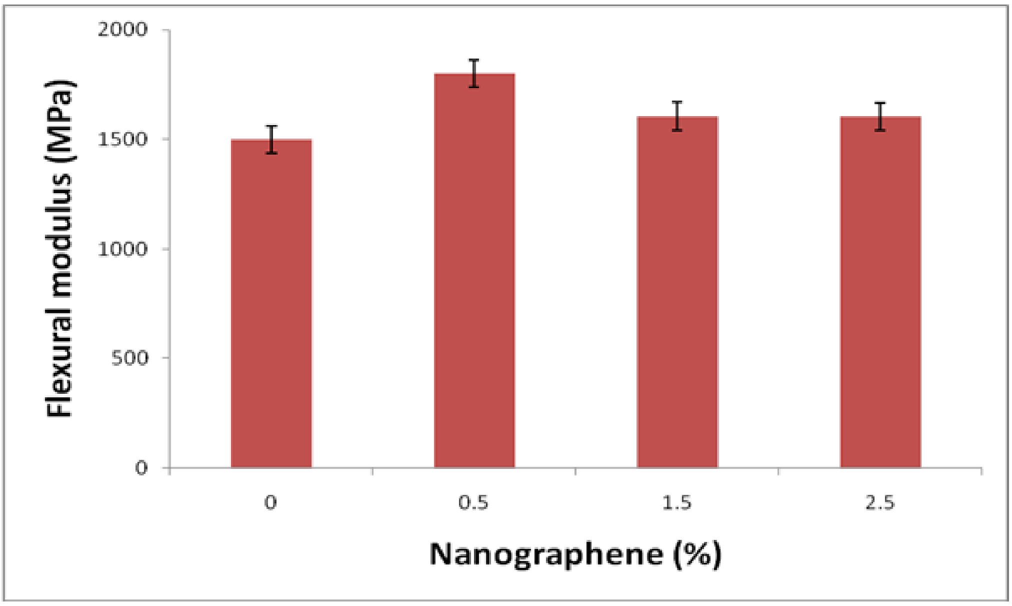

Fig 2: Nanographene loading vs. flexural modulus of FPF/HDPE nanocomposite

Changes in the flexural modulus of composites made of FPF/HDPE with different levels of nanographene loadings are shown in Figure 2.The highest (1800MPa) and lowest (1500MPa) average flexural modulus values were obtained for the samples with 0.5 wt % and 0 wt % nanographene respectively. Increasing the amount of nanographene to 0.5 wt % caused the flexural modulus to increase and thereafter further increase resulted to a decrease in flexural modulus. By increasing nanographene loading to 0.5 wt % in epoxy resin, the modulus of elasticity increased; with further increase up to 2 wt % it is reduced [28]. In another study, Sheshmani *et al*., [9] reported that the highest modulus of elasticity was obtained at 0.8 wt % graphene loading, and after that higher percentages lowered the modulus.

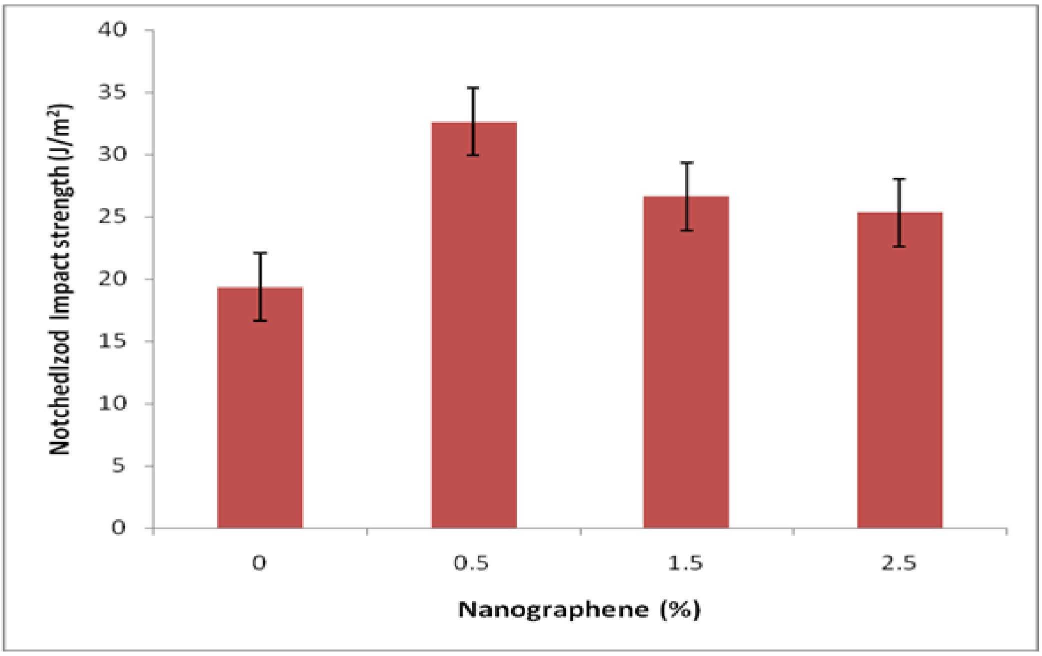

Fig 3: Nanographene loading vs. Impact strength of FPF/HDPE nanocomposite

Variations in notched impact strength of FPF/HDPE composites at different levels of nanographene loading are depicted in Figure 3. The highest (32.61J/m^2) and lowest (19.35J/m^2) average notched impact strength were observed in samples with 0.5 wt % and 0 wt % nanographene respectively. By increasing nanographene to 0.5 wt %, the notched impact strength of FPF/HDPE composites increased and then decreased to the level of 2.5 wt %. The reduction in impact strength was predictable because nanographene particles caused more embrittlement of composites and reduced their impact strength. Nanoparticles in the polymer matrix reduced mobility and possibility of their energy loss, increasing the energy absorbed by the composite and creating spots of stress concentration. These spots initiate cracks and failures [29].

Morphological properties

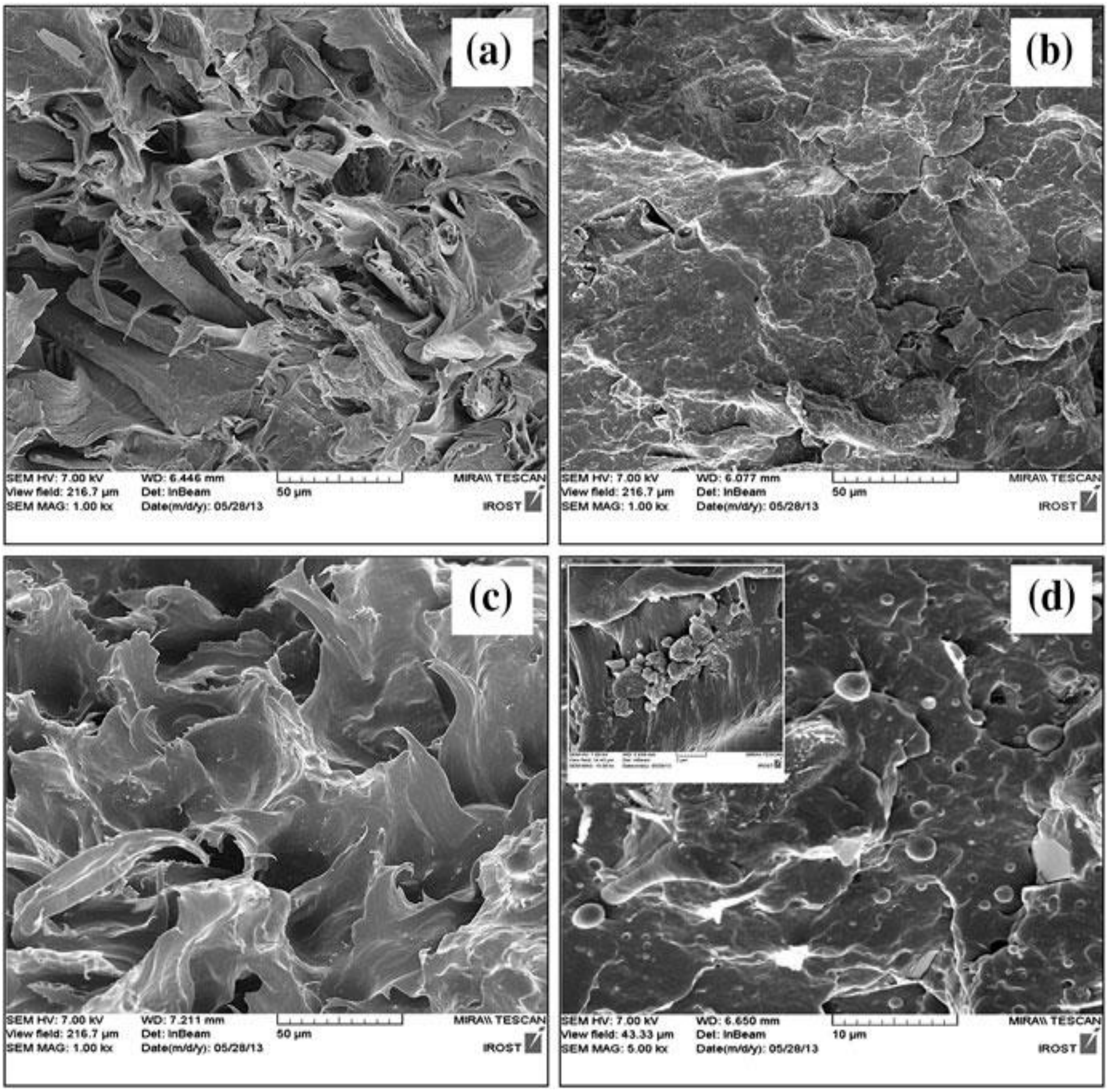

Fig 4: SEM images of composites containing 35 wt % FPF (a) 0 (b) 0.5 (c) 1.5 (d) 2.5% nanographene

The significant improvement in mechanical properties of the composites with incorporation of nanographene is well evidenced by SEM micrographs. Through SEM study, the distribution and compatibility between the filler and the matrix could be observed. Figure 4a corresponds to FPF/HDPE without nanographene (control sample), showing some evidence of fiber pull-out from matrix. Therefore, when stress is applied it causes the fibers to exit the matrix easily and causes micro voids. As seen from Figure 4b (containing 0.5 wt % nanographene), there is no separation of the fibers from the matrix and a very good interaction between the components can be interpreted from the image. The strong adhesion that is observed at the interface has been already discussed in mechanical properties of the composites and is related to encapsulation of fibers in the HDPE matrix, which causes strong bonding. The surface of composites containing 1.5 wt % nanographene is depicted in Figure 4c. There are some cavities in FPF/HDPE composite that can absorb water and/or reduce mechanical properties. As illustrated in Figure 4d high content of nanographene was easily agglomerated, which is the characteristic of this nanofiller. In addition, Figure 4d shows the position of agglomerated nanoparticles in the composites with 2.5 wt % nanographene. The presence of these agglomerates results in the generation of flaws and subsequent creation of voids between the filler and the matrix polymer. This causes the mechanical properties of the composites to be reduced, as compared with the composites filled with lower content of nanographene [29].

CONCLUSIONS

The effect of nanographene addition on the mechanical and morphological properties of fluted pumpkin stem recycled high density polyethylene nanocomposites has been investigated. From these investigations, the following conclusions were drawn: Incorporating nanographene into the polymer matrix significantly improved the flexural strength, flexural modulus and notched impact strength of the FPF/HDPE nanocomposites and this enhancement came at a unique nanographene loading of 0.5 wt %. Increasing the nanographene loading above 0.5 wt % brought about a reduction in the mechanical properties of the nanocomposites as evidenced by Scanning electron microscopy.

Declaration of Conflicting Interests

The author(s) wish to proclaim that there is no potential conflict of interest as it relates to the research, authorship, and publication of this article.

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