Nigerian Journal of Textiles (NJT)
A Publication of Textile Researchers Association of Nigeria (TRAN)
Volume 7 August 2021
http://tran.org.ng

Fabrication And Characterization of Flambouyant Seed Particles-filled Unsaturated Polyester Composites

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ABSTRACT
The research aimed at fabrication and characterization of Flambouyant Seed Particles (FSP) filled unsaturated polyester (UP) composites. The composites fabrication was achieved via hand layup with particle size of 100 µm at different filler loading of 2, 4, 6, 8, 10, 12, 14, 16, 18, 20 and 22 wt %. The composites were characterized by tensile properties and density. The result showed an increase in tensile strength, tensile modulus, and density as the filler loading increased to optimum values of 37.02 MPa, 0.47 GPa and 1.25 g/cm^3 respectively, and afterwards decreased with further increased filler loading. The % elongation at break increased on the addition of the filler to the matrix and marginally decreased as the filler loading increased. The optimum and minimum values of % elongation at break were 8.15 % and 5.46 % at 2 and 22 wt % respectively. The scanning electron micrograph of the 10 wt % sample shows better interaction between the FSP and the matrix when compared to the 20 wt % sample, with clear failure that indicates weak interfacial adhesion. Thus, improved interfacial interaction enhances the material's mechanical properties.

Keywords: *Density, Flambouyant seed particles, filler loading, tensile properties and unsaturated polyester*

INTRODUCTION

The increase in environmental consciousness and community interest, the new environmental regulations and the unsustainable petroleum depletion led to thinking of new environmentally friendly materials. Nowadays, cellulosic fibres are considered an effective material for composites reinforcement due to increased global environmental awareness and new environmental regulations, sustainability, and societal concerns have given a continuous approach to the ecological design of environmentally friendly composites (Low, 2018). Several cellulosic products and wastes such as shell flour, wood flour and pulp are used as fillers in polymers (Jawaid and Abdul Khalil, 2011). The motivation to introduce natural reinforcement to a matrix is to cut the production cost, encourage waste reusability and turn it into a value-added product (Kuan *et al.*, 2021). Solid wastes produced from agricultural activities are one of the sectors generating a reasonable amount of waste, which may be allowed to accumulate indiscriminately and constitute a nuisance to global health or used as raw materials for bio-economy (Adejumo and Adebiyi, 2020).

Agricultural waste usually disposed into the environment causes a menace to the community either by littering, dumping or burning, such as flambouyant pods that dropped from the plants when matured as by-products. The waste bio-mass not effectively used in composites reinforcement includes flambouyant pod. Flambouyant plants, known as flame plants, are a natural resource whose potential as filler reinforcement in polymer composites has not been utilised. Its pods obtained from the flame plants are agricultural wastes that are abundantly available, renewable resources and biodegradable. Its seed particles can be used as reinforcing materials for unsaturated polyester. Therefore, producing natural filler reinforced unsaturated composites that are cost-effective and have superior properties over their synthetic fibres. Wilson García *et al.,* (2021) suggested that agricultural waste is an option for reinforced plastic composites due to its low density, high mechanical resistance, low cost, biodegradability, non-toxic and high availability. This study aimed at exploiting flambouyant seed particles as an effective means of controlling waste biomass (flambouyant seed) via producing value-added composites.

MATERIALS AND METHODS

Materials

The raw materials used for the composites fabrication includes Unsaturated Polyester (UP), a catalyst; Methyl Ethyl Ketone Peroxide (MEKP), promoter (cobalt naphthenate), and flambouyant

Seeds Particles (100 µm) (FSP). Equipment used include glass moulds (dimensions: 100 mm x 100 mm x 3 mm), a Retsch Masch 70992 jaw crusher, a ball mill grinding machine (Kera b.v. 057748), and a standard sieve (Seive-Tronic ISO 3310-1:2000, BS 410-1:2000).

Methods

Sample Preparation

The flambouyant pods were split open, the seeds collected, crushed using a jaw crusher (Retsch mesh Nr.70992) and the hard seed coat (testa) separated from the soft inner part (endosperm/cotyledon). The coats collected were washed with clean water to remove all the endosperm/cotyledon particles, air-dried for 48 hours, and then oven-dried at 60 ºC for 48 hours to ensure proper drying. The dried seeds were ground with a ball mill grinding machine (Kera b.v. 057748) and then sieved to particle size 100 µm using a standard sieve (Sieve-Tronic ISO 3310-1:2000, BS 410-1:2000).

Composites Preparation

Composites were prepared using different filler loading of 2, 4, 6, 8, 10, 12, 14, 16, 18, 20 and 22 wy %. The mould for each casting was properly scraped, cleaned, and well lubricated with wax and then placed on it a foil paper. The quantity of UP required for each sample was measured and poured into a mug, and the required filler loading was also measured and then added and mixed. The catalyst, MEKP (1 wt. %), was added and stirred for 2 minutes, followed by the addition of 1 wt. % of the promoter (cobalt naphthenate) and stirred for an additional 3 minutes. The mixture was then carefully poured into the mould, covered with another glass sheet of the same size, and then pressed for proper matrix distribution. The samples were cured under pressure using a load at ambient temperature for 24 hours. The composites were later removed from the mould and cut into dumbbell dimensions.

The Tensile Test (ASTM D638)

The tensile test of the sample composites was according to ASTM D638 using a tensile testing machine (model: TM 2101 – T7) with a maximum force of 10 KN and a cross-head speed of 2 mm/min at ambient temperature of 25±3 ºC. The sample dimensions were determined using vernier calliper. The test were held in the grips of the testing machine and tightened evenly and firmly to prevent any slippage as the test commenced. The uni-axial load was applied to each end of the respective samples until they failed. The resistance and elongation at break of the specimen were detected and recorded. The tensile properties were obtained from the stress-strain curves plotted via the force-extension data on a special graph paper during the test. The properties (tensile strength, tensile modulus, and elongation at break) were determined and recorded.

Density Measurement (ASTM D792)

The density of the composites determined according to ASTM D792. The mass of each composite sample was determined using an analytical weighing balance, and the volume obtained via the dimensions of each side (length x breadth x width) accurately measured using a digital vernier calliper. The density of the sample was computed as the ratio of mass to volume (g/cm^3).

The bulk density of the particles is determined by ASTM D7481-18, Method A. The FSP sample was conveyed into a graduated cylinder and the mass obtained. The bulk density of particles was computed as the ratio of the mass of an untapped particle sample and its volume (g/cm^3).

Morphological Property

The tensile fractured specimens were used to study the surface morphology of the composites using a scanning electron microscope (SEM, Joel TM, model JSM-7600F) operated at 20 KV and a magnification of 8000X.

Results and Discussion

The Effect of Filler Loading on Tensile Properties

The tensile strength and modulus of the composites are shown in Figure 1. The result showed that with the addition of the filler, the tensile strength of the composite increased from 27.55 MPa to 32.40 MPa at the corresponding filler loading 0 and 2 wt. %, and then gradually increased to a maximum value of 37.02 MPa at 10 wt. % respectively and afterwards decreased gradually to 24.67 MPa at 22 % filler loading. The increase in the tensile strength is attributed to the improved interaction between the filler and matrix, which improves the interfacial area and contributes to good interfacial bonding between the hydrophilic filler and hydrophobic matrix. The increased tensile strength with higher filler loading indicates good compatibility of the natural filler with the matrix and improved interface strength (Zahari *et al*., 2015). More compact bridges form when porosity

decreases and the obliteration of inter-particle forces become more difficult (Eichie and Kudehinbu, 2009). The increase in tensile strength with an increase in filler loading is in agreement with the findings of Chow *et al*., (2020); Daramola *et al*., (2017), Onuoha *et al*., (2017); Nwanonenyi *et al*., (2013); Ameh *et al*., (2015); Njoku *et al*., (2011) and Onuegbu and Igwe, (2011).

The effect of filler agglomeration acts as an obstacle to filler dispersion, increases with an increase in filler loading which consequently lowers the filler-matrix interactions and results in to decrease in tensile strength after the optimum amount of filler has been added (Ekwueme and Igwe, 2018). This trend of increase in tensile strength to an optimum value and afterwards decreases with an increase in filler loading was similarly observed in some research findings (Zakari *et al*., 2020; Ekwueme and Igwe, 2018; Kucukdogen *et al*., 2016; Abdulrahman and Haruna, 2015; Ameh *et al*., 2015; Zahari *et al*., 2015).

The tensile moduli of the composites observed in the figure showed higher tensile modulus of 0.43 GPa before the incorporation of filler (0 % filler loading). The result indicates that the polymeric composite material is less stiff. The presence of the filler within the matrix made the composite stiffer, leading to a decrease in the strength of the composite (Nwanonenyi and Chike–Onyegbula, 2013). A decrease in the tensile modulus of composites after the initial addition of fillers to the matrix is due to poor mixing or uneven filler dispersion in the polymer matrix (Okey-Onochie *et al*., 2020). Thereafter, the modulus increased steadily with increased filler loading from 0.38 GPa at 2 wt % to an optimal value of 0.47 GPa at 12 wt. %, and afterwards, the modulus gradually decreased with an additional increase in filler loading to 0.35 GPa at 22 wt. %. Similar tensile moduli obtained for both 2 and 4 wt. % filler loading with a value of 0.38 GPa and 14 % and 16 % filler loading with a value of 0.42 GPa. The composites modulus increased with an increase in particle filler loading into the polymer matrix signifying good interfacial bonding between the filler and the matrix. The fillers incorporated into the polymer matrix improve the composite's rigidity and the properties of the matrix (Nwanonenyi *et al*., 2013; Onuegbu and Igwe, 2011). Some studies have reported a similar trend of increased tensile modulus with the increase in filler loading (Hanana and Rodrigue, 2020; Ekwueme and Igwe, 2018; Daramola *et al*., 2017; Onuoha *et al*., 2017; Kolawole *et al*., 2015; Zahari *et al*., 2015; Nwanonenyi *et al*., 2013; Onuegbu and Igwe, 2011; Rahman *et al*., 2010).

The comparable trends of decreased tensile modulus of composites with the increase in filler loading were also reported (Wilson Garcia *et al*., 2021; Okey-Onochie *et al*., 2020; Manjunatha and Ahmed, 2017; Ameh *et al*., 2015; Nwanonenyi and Chike–Onyegbula, 2013). The decrement in tensile modulus shows poor resistance of a material to deformation because, at high filler loadings, the composite will not be able to withstand greater loads (Nwanonenyi and Chike–Onyegbula, 2013). The reduction may be due to the interfacial voids which act as stress concentrators or the incompatibility of the fillers with the matrix (Okey-Onochie *et al*., 2020). This irregular pattern of tensile modulus of elasticity was consistent with some reports (Daniel *et al*., 2017; Abdulrahman and Haruna, 2015).

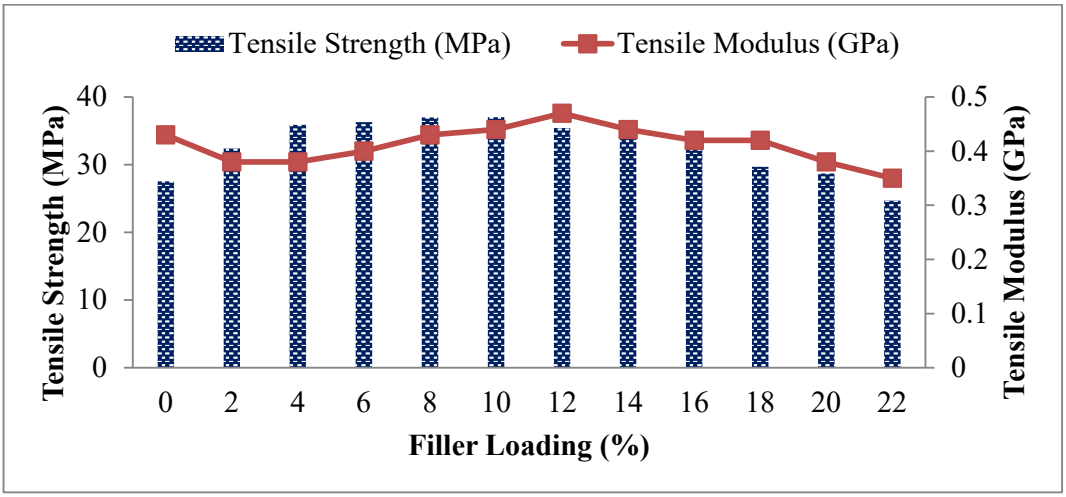

Figure 1. Effect of Filler Loading on Tensile Strength and Modulus of UP-FSP Composites

The Effect of Filler Loading on Elongation at Break

The percentage (%) elongation at break of the composites is shown in Figure 2.

The graph showed an increase in % elongation at break by addition of the fillers to the UP matrix. The increase may attribute to the uneven distribution of the FSP within the matrix.

The low extent of homogeneity of the mixture may be responsible for the increased % elongation at break (Abdulrahman and Haruna, 2015). Afterwards, the % elongation at break decreased marginally as the filler loading increased. The optimum and the minimum % elongation at break of 8.15 % and 5.46 % obtained at 2 and 22 wt. % respectively.

The decrease ductile in elongation at break with increasing filler loading attributes to the increased stiffness, which lead to a reduction in elasticity of the UP matrix resulting in poor stress transfer from the fillers to the matrix.

A lower value of % elongation at break indicates stiffness of the material (Manjunatha and Ahmed, 2017). It also shows the inability of the filler to support the stress transfer from the matrix to particle fillers (Okey-Onochie *et al*., 2020; Abdulrahman and Haruna, 2015; Nwanonenyi and Chike–Onyegbula, 2013).

The increase in stiffness of the composite with the increase in the filler loading is due to an increase in the deformation of a rigid interfacial interaction between the fillers and matrix, and it directs the composite to incline towards loss of strength and weakness of a material (Nwanonenyi *et al*., 2013).

It also resulted in the stiffening and hardening of the composites, thereby reducing their resilience and toughness and leading to lower elongation at break (Okey-Onochie *et al*., 2020). The observed decrease in elongation at break of the composites with increased filler loading is consistent with previously reported works on the variation of % elongation at break with filler loading of natural fibre reinforced composites (Okey-Onochie *et al*., 2020; Sahadat Hossain *et al*., 2020; Ekwueme and Igwe, 2018; Manjunatha and Ahmed, 2017; Onuoha *et al*., 2017; Abdulrahman and Haruna, 2015; Zahari *et al*., 2015; Nwanonenyi and Chike–Onyegbula, 2013; Nwanonenyi *et al*., 2013; Onuegbu and Igwe, 2011).

Effect of Filler Loading on Density

The density of the composites is shown in Figure 3. The results show that the density of the 2 wt. % loaded composite is lower than that of the neat UP (0 wt. %) composite.

The dropped in density attributes to the lower density of the FSP (bulk density ≈ 0.35 g/cm^3) relative to UP with a higher density (≈ 1.17g/cm^3). Researchers in their studies observed similar decrease in density with the incorporation of filler into the matrix (Ameh *et al*., 2015; Kolawole *et al*., 2015).

The result showed a progressive increase in density with increased filler loading to an optimum value and thereafter decreased with a further increase in filler loading. The maximum density value of 1.25 g/cm^3 observed at 18 % filler loading.

Researchers reported similar trends (Das and Biswas, 2016; Ameh *et al*., 2015). Hanana and Ridrigue, (2021) studied the effect of particle size, fibre content, and surface treatment on the mechanical properties of maple-reinforced low linear density polyethene produced by rotational moulding, reported an increase in density with increased filler loading.

The decrease in density with increased filler loading attributes to poor distribution and filler agglomeration in a polymer matrix. Wilson Garcia *et al*., (2021) reported that a decrease in density with an increase in filler loading is due to random dispersion and agglomeration of the fibre in the matrix.

Morphological Studies

The SEM micrographs of the tensile fractured surface are shown in Figure 4.

The micrograph (Figure 4a) shows a fairly regular dispersion and bonding between the FSP and the unsaturated polyester at 10 wt % filler loading. This shows good interfacial interaction between the filler and UP matrix, which contributes to its improved mechanical properties.

At 20 wt % filler loading (Figure 4a), the interface between the particles and the unsaturated polyester is poor due to the restriction of unsaturated polyester chain mobility and the uneven distribution of the particles in the matrix caused by the increase in particle concentration. Thus, poor distribution leads to the poor mechanical properties of the composite.

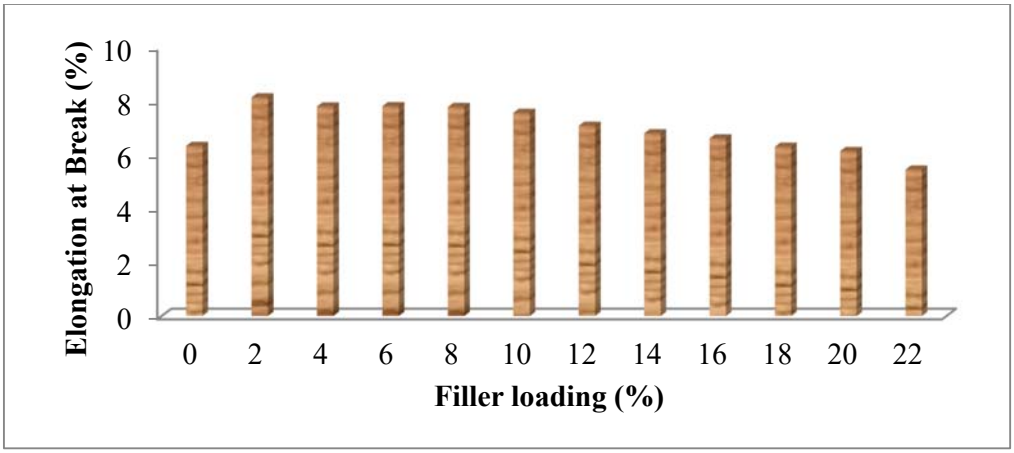

Figure 2. Effect of Filler Loading on % Elongation at Break of UP-FSP Composites

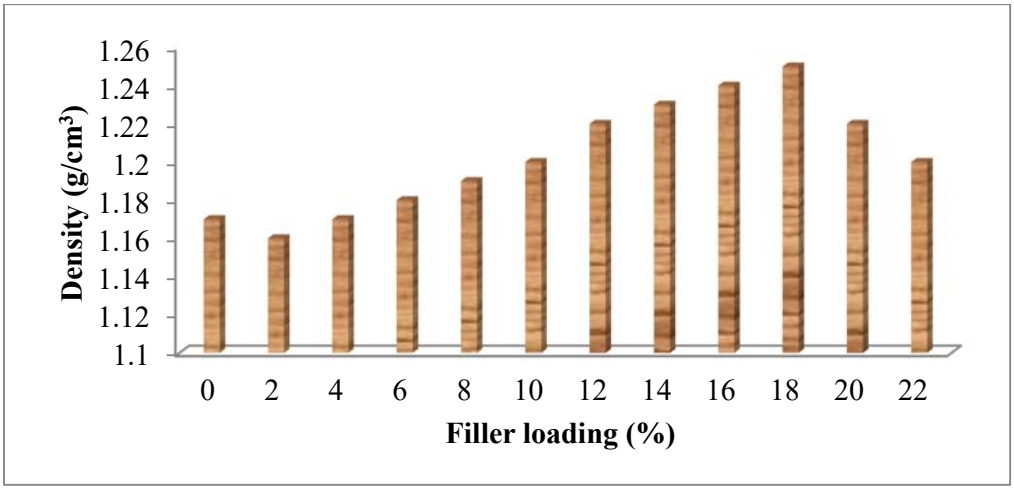

Figure 3. Effect of Filler Loading on Density of UP-FSP Composites

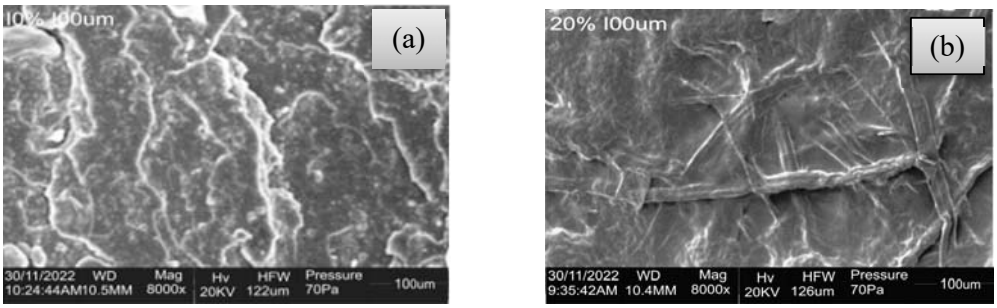

Figure 4: SEM Micrographs of fractured surface of UP-FSP Composites (a) 10 wt % and (b) 20 wt % filler loadings at a magnification of 8000X.

Conclusion

The study successfully prepared UP-FSP composites. The results showed that the tensile strength, tensile modulus and density of the composites increased as the filler loading increased to corresponding optimal values of 37.02 MPa, 0.47 GPa and 1.25 % at filler loading of 10, 12 and 18 wt % respectively, and afterwards, decreased with increased filler loading. The SEM micrographs of the tensile fractured surface show a fair interfacial interaction between the filler and the UP at 10 wt % when compared to 20 wt % with poor interface. The results showed that the composites fabricated can be used in low-load bearing applications.

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