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ORIGINAL RESEARCH ARTICLE

PRODUCTION OF BIOCOMPOSITE FROM Parinari polyandra FRUIT SHELL USING WASTE LOW DENSITY POLYETHYLENE AS MATRIX

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ARTICLE	ABSTRACT		
INFORMATION	Enormous w		
Submitted 20 October, 2021 Revised 22 February, 2022 Accepted 3 March, 2022	polyethylene (in Nigeria. Th LDPE. Bioco Parinari polyo – loading (10%		
Keywords: Biocomposites <i>Parinari polyandra</i> low-density polyethylene matrix	fabricated for were charact strength test, spectroscopy composite mi revealed that size 0.42mm of 320.321 N for compress Flexural, impo		

astes that are continuously being generated from low-density (LDPE) film sachet water have remained an environmental challenge hese might require urgent action due to the non-biodegradability of proposite samples were produced using waste LDPE sachets and andra shells, where filler sizes (0.420 mm, 0.841 mm) and filler 5, 20%) were varied. In this study, a melt mixer was designed and r the production of bio composite samples. Biocomposite samples terized using tensile tests, compression test, flexural and impact water absorption test, biodegradability, fourier transform infrared and scanning electron microscopy tests. Viscosity of the bio ix was a major constrain in the design of the melt mixer. The results the optimum value of tensile strength was obtained with particle at a 10% filler to matrix loading which produced a Young's modulus N/mm^2 . Higher particle size and filler loading elicited a higher value vive strength based on a force at peak value of 27552.500 N. act and tensile strength decreased with increasing filler loading and particle size, while increased immersion time gave increased percentage of water absorbed.

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1.0 Introduction

Enormous waste generated from low-density polyethylene (LDPE) packaged sachet water constitutes an environmental challenge in Nigeria which demands attention due to its nonbiodegradability as attested by several studies (Chatterjee et al., 2010; Sylvester et al., 2020). With the recognition that landfill is not sustainable and its costs are increasing, waste disposal becomes important with more responsibility being placed on producers. Governments of several countries have implemented laws to encourage the use of recycled and/or bio-based 'green' products (Suheyla et al., 2016).

Composites are multifunctional materials with exceptional mechanical and physical properties which can be custom fitted to meet the prerequisites of a specific application (Badamasi and Ibrahim, 2017; Rubentheren et al., 2015). Composite materials comprising of one or more phases derived from a biological origin are referred to as biocomposites (Fowler et al., 2006). In general, the main components of bio composite materials are fibers and matrix. The benefits of plant fibers are acceptable specific strength, low cost, low density, good thermal insulation properties, reduced dermal and respiratory irritations, renewable resource and possible recycling without

affecting the environment (Busuioceanu *et al.*, 2018). The matrix binds the fibers together, allowing the transfer of tension between the fibers in the composite.

Parinari polyandra Benth belongs to the Rosasceae family, it is also known as Maranthes polyandra Benth, it is a grassland plant found in West Africa (Abolaji et al., 2007). Parinari fruit shells are wastes generated from Parinari polyandra Benth seed oil utilization. While many studies have been carried out on utilization of natural fillers and fibers such as short silk fibers, kenaf, sisal, jute, oil palm, empty fruit bunch, coir, sugar palm, rice husk ash, and coconut shell powder, little work has been reported on the use of Parinari fruit shell as fillers in polymer matrices. Thus, this work investigates the properties of biocomposites produced from waste low-density polyethylene sachet and Parinari polyandra shell, thereby fostering the conversion of wastes to value-added products which can be used for wider applications commercially.

I.0 Materials and Methods

2.1 Materials used

Parinari fruits were harvested from its trees within the campus of University of Ilorin, Kwara State, Nigeria. Harvested fruits were cut in half to separate the seeds from the shells, after which shells were cleaned and sundried. The clean dried shells were size reduced using a grinding machine and screened to sizes 0.420 mm and 0.841 mm. LDPE waste water sachets were sourced and collected from restaurants and shops within the University of Ilorin campus. Waste LDPE sachets were washed to remove oil and dirt after which they were dried and shredded to give better surface area for heating. Base oil was sourced from LUBCON Oil, Ilorin and used as lubricant for mould edge lubrication. Figures I and 2 show the image of *Parinari polyandra* shells and waste LDPE respectively.



Figure 1: Parinari polyandra Shells



Figure 2: Waste LDPE

Equipment used in this research were: Pulverizing/grinding machine, Testomeric universal Testing Machine FS50AT, Shimadzu Fourier transform infrared spectrophotometer – FTIR 8400 S, Avery Denison 150J Impact Testing Machine 6705U/33,122, screening mesh sizes of 0.42mm and 0.841mm, fabricated metal moulds with dimensions 200×20×10 mm³, 50×50×50 mm³, 40×10×10mm³ and 200×25×15mm³ for tensile, compression, impact and flexural strength respectively, weighing scale, shredder/scissors and a fabricated melt mixer. Figure 3 shows the Testomeric universal Testing Machine FS50AT.



Figure 3: Testomeric Universal Testing Machine

2.1.1 Mixer material of construction and geometry

The melt mixer consists of a fabricated cylindrical shaped 2 mm stainless steel tank, unbaffled with diameter 300 mm and height 400 mm. This was scaled up based on the required mass of (500 g) for batch mixing which puts into consideration a mix of shredded waste low density polyethylene sachets and size reduced *Parinari* shells. The relatively high cost and unavailability of a row mill influenced the initiative to design and fabricate a melt mixer using available local resources.

2.1.2 Impeller blade construction and geometry

Standard shape factor requires the diameter of the impeller blade to be one-third the diameter of the tank and same ratio for the clearance off the base of the tank.

i.e
$$D_a = \frac{1}{3}D_t$$

(1)

where D_a: Diameter of impeller blade (mm)

D_t: Diameter of tank (mm)

To ensure an efficient molten mix it was imperative to have a very small clearance off the base of the tank hence, a paddle shaped impeller blade at the base with a 45° inclination tip and a 270 mm diameter was used because it thrives better for conditions of high viscosity and fits closely to the base of the tank.

2.1.3 Lagging material

Fiber glass was the lagging material used, this was done to retain heat within the melt-mixer enhancing an adiabatic condition.

2.1.4 Heat transfer requirement for heating coil/element

Heating coil was wired round the entire fabricated tank to achieve uniform temperature at all sections of the tank. Considering the melting point of shredded waste low density polyethylene of 120 °C (Osuolale and Agbalaya, 2012), the temperature range was selected as 27 - 120 °C, specific heat for low density polyethylene film ranges between 1900 and 2300 Jk⁻¹kg⁻¹. In calculating heat transfer requirement for the heating coil;

q = mcdt

Where;

q = heat transfer (J)m = mass of substance (kg)c = specific heat (J/kg. k)dt = Temperature difference (°C)q = 106,950 Jdt = Temperature difference (°C)

(2)

Heating element with 1500W was used in the fabrication process which had the capacity to provide a temperature range from 30° C to 300° C

2.1.5 Power requirement for mixer

Power = $N_p \times n^3 \times d^5 \times S_g \times K$ (3) Where; N_p = Power number n = Rotation speed (r/s) d = Diameter of impeller (mm) S_g = Specific gravity ($K_g m^{-3}$) K = Reynolds number Impeller Diameter D = 270 mm = 0.27 m S_g for molten LDPE = 760 $K_g m^{-3}$ $n = 70 rpm = \frac{70}{60} r/s$

$$R_{e} = \frac{nd^{2}\rho}{\mu_{a}}$$

where $\mu_{a} = 1.12 \times 10^{5}P = 1.12 \times 10^{2}P_{a}$. S

Which is the 0 shear rate viscosity for Linear LDPE whose value by assumption will be close and used for that of molten LDPE

 $R_{e} = 0.577$

therefore $N_p = 40.98$ extrapolated from power correlation for a six blade

turbine for pseudo – plastic liquids

P = 40.94 W

A DC variable motor of 1 horse power was used in fabrication which provided a 10-70 rpm range.

2.1.6 Vent

A vent or smoke outlet at the top of the vessel was used to help channel smoke formation.

2.1.7 Molten outlet

A ball valve tap outlet from where the bio composite molten mix can be removed to be placed into desired moulds.

2.2 Method

Waste low density polyethylene was melted thermally i.e. by application of heat. The polymer melt was then mixed thoroughly with filler. The factors, filler size and filler-matrix ratios were varied one after the other. Filler sizes varied were 0.420 mm and 0.841mm while the varied filler to matrix ratios were 10% and 20%. The waste LDPE – *Parinari* shell composite were mixed in the ratios 10/90, 20/80 till a uniform mix was achieved. Each composite mix was transferred into fabricated metallic moulds of different dimensions based on specific tests to be carried out. Prior to mould placement, base oil was applied to the fabricated mould edges to enhance the ease of

(4)

bio composite removal after bio composite samples had solidified. Hand lay-up technique was used (Hussein, 2013). Table I shows how the factors were varied one at a time.

RUN	Filler Size (mm)	Filler-matrix ratio (%)
1	0.42	10
2	0.42	20
3	0.84	10
4	0.84	20

Table I: Experimental runs varying one factor at a time

2.3 Characterization

2.3.1 Flexural strength

Flexural strength test was carried out on bio composite samples with mould dimension of 200 mm \times 25 mm \times 15 mm using a universal testing machine FS50AT.

2.3.2 Compression test

Compression test was carried out on produced bio composite with mould dimension 50 mm×50 mm × 50 mm using a universal testing machine FS50AT.

2.3.3 Tensile Strength

Tensile strength was measured using a universal testing machine FS50AT with test specimen dimensions according to ASTM standards until test failure occurred. Mould dimension used for test was 200 mm \times 20 mm \times 10 mm where values for stress at peak, force at peak, and Young's modulus were obtained (Zaaba et al., 2013).

2.3.4 Scanning electron microscopy

Scanning electron microscopy was used to assess the morphological characteristics of selected bio composite produced. The test was done to show the uniformity and adhesion of filler and matrix. The scanning electron micrograph was shown with a (500x) magnification.

2.3.5 Fourier transform infrared spectroscopy

Infrared spectroscopy was performed using a Shimadzu Fourier transform infrared spectrophotometer – FTIR 8400 S. Samples were scanned in the absorbtion area of 500 to 4000 cm⁻¹ where transmittance values ranged from 0 to 100%. IR peaks were observed in correspondence with their bond types.

2.3.6 Water absorption

Water absorbed by biocomposites produced was determined by drying and immersing the samples in distilled water, samples were weighed every day for four days to obtain the new change in weight. The percentage water absorption was then calculated using the formula (Obasi, 2015).

%water absorbed =
$$\left[\frac{final \, dry \, weight \, after \, immersion}{initial \, dry \, weight \, before \, immersion} - 1\right] x \, 100$$
 (5)

2.3.7 Impact test

Impact test was used to test for the impact strength of materials. The test was carried out using the Avery Denison 150J Impact Testing Machine 6705U/33,122. The impact test was conducted on notched rectangular samples with dimensions according to ASTM D256.

2.3.8 Biodegradability test

A two-week biodegradability test was performed for produced bio composite samples. Biocomposite samples were buried in the soil and unearthed for 7 days, dried, weighed, buried again and unearthed after another 7 days and weighed again. (Junior *et al.*, 2015).

3. Results and Discussion

3.1 Melt mixer design and fabrication

Chemical engineering design and AUTOCAD design of the melt mixer was achieved. Fabricated melt mixer was successful with heating and mixing of polymer melt and parinari shell particles. Figures 4 and 5 show the AUTOCARD design and the fabricated view of the melt mixer.



Figure 4: Wireframe AutoCAD design view

Figure 5: Fabricated melt-mixer

3.2 Flexural Stress

Figure 6 shows that stress at peak for filler size 0.420 mm decreased with increasing filler loading from a value of 16.0022 N/mm² to 9.748 N/mm² while the bending modulus increased from a value of 312.594 N/mm² to 410.129 N/mm² as observed on Figure 7. Filler size 0.841 mm had a slightly differing trend such that the stress at peak increased with increasing filler loading from a value of 6.028 N/mm² to 16.064 N/mm² while having same trend for bending modulus where an increase was observed from 245.219 N/mm² to 567.381 N/mm².

It was observed that bending modulus increased with filler loading for both filler sizes, particle size 0.841mm with 20% filler loading had the highest value while particle size 0.841mm with 10% filler loading had the lowest value. Saliu *et al.* (2015) observed flexural strength decreasing with increasing filler loading, this occurred due to insufficient wetting of incorporated fibers by matrix. Fiber-fiber adhesion in fiber bundles reduces contact area between fiber and matrix which causes poor stress transfer from matrix phase to dispersed fibers (Moh'd Ishak *et al.*, 1997).

As fiber loading kept increasing the modulus also increased, this probably verifies the increase in the modulus of biocomposites produced. This is found to be in agreement with a study conducted by Dhal and Mishra (2013); Ku *et al* (2010).



Figure 6: Flexural stress varying with filler to matrix ratio and size



Figure 7: Bending modulus against filler to matrix ratio and size

3.3 Compressive strength

Compressive strength is the capacity of a material to withstand loads tending to reduce size. Table 2 shows that with increasing filler content for filler size 0.420mm, strain at peak, force peak and Young's modulus show reduction in value, while with increasing filler content for filler size 0.841mm increase is observed for both strain at peak and force at peak, and a decrease in value for young's modulus. The highest force at peak was observed for particle size 0.841mm with 20% filler loading as observed on Figure 8, highest young's modulus 107.263 N/mm² is observed for particle size 0.841 with 10% filler loading.

Using force at peak as a yardstick for compressive strength, 0.841mm with 20% filler loading will prove viable in that it is in conjunction with approximately a 38% strain. So we have a higher particle size with higher filler loading eliciting a higher value for compressive strength based on a force at peak value of 27552.5N.

Size (mm)	Filler to Matrix	Strain at peak (%)	Force @ Peak (N)	Young's Modulus
	Ratio (%)			(N/mm²)
0.420	10	27.1	24110.1	98.95
0.420	20	26.9	19810.9	87.98
0.841	10	25.1	23697.2	107.26
0.841	20	38.2	27552.5	75.05
0	0	23.6	22654.2	100.2





Figure 8: Force at peak against filler to matrix ratio and size

3.4 Tensile strength

Filler size 0.420mm with filler to matrix ratio 10% has the highest stress at peak value of 7.366 N/mm² while filler size 0.841mm with filler to matrix ratio 10% and 20% have lower stress at peak values based on filler and matric mixed, but nothing compared to 100% waste low density polyethylene matrix as seen on Table 3. The highest young's modulus value is observed with filler size 0.420 mm and its ratio 20% on Figure 10. Since tensile strength is the maximum load a material can support without fracture when being stretched per the original cross-sectional area of the material, maximum load 1252.300 N makes filler size 0.420 mm with filler to matrix ratio 10% bio composite sample with the highest tensile strength.

An increase in filler size and loading reduced tensile strength, this is graphically illustrated on Figure 9. Al-assafi *et al.* (2016) experienced a drop in tensile strength with increasing nanofiber content, Gumel *et al.* (2014) observed the same trend where the addition of filler interfered with regular arrangements of rubber molecules used as matrix, same can be said for waste low density polyethylene molecules being interfered by more addition of filler which is a probable cause for the reduction in tensile strength where filler - matrix interaction is reduced.

Size (mm)	Filler / Matrix Ratio (%)	Stress at peak (N/mm²)	Young's Modulus (N/mm²)	Force at peak (N)	Area
0.42	10	7.4	320.3	1252.3	170
0.42	20	3.92	432.2	665.7	170
0.84	10	3.53	348.1	600.6	170
0.84	20	3.86	290.1	656.3	170
0	0	9.78	142.4	141.98	170





Figure 9: Tensile stress at peak against filler to matrix ratio and size





Figure 10. Young's modulus against filler to matrix ratio and size

3.5 Scanning electron microscopy

Scanning electron microscope micrograph shows the fiber-matrix interaction and surface morphology of the produced bio composite sample with particle size 0.42 mm and 10% filler loading. Figure 11 shows the fiber-matrix interactions gotten through matrix wetting the fillers. From the Micrograph image, areas with good adhesion can be observed which explain areas with good uniformity and can be traced to fibers being sufficiently wetted by the waste low density

polyethylene matrix. Some cracks and voids can as well be observed on the micrograph image showing areas with reduced adhesion and uniformity, weak interfacial adhesion can be a probable cause (Zaaba *et al.*, 2013).



Figure 11: Scanned micrograph for particle size 0.42mm, 10% filler loading

3.6 Fourier transform infrared spectroscopy

Figure 12 shows the FTIR spectra for particle size 0.42mm with 10% filler loading. FTIR analysis helps to pinpoint a sample's molecular composition and structure, it identifies the chemical bonding in molecules. IR peaks on Figure 12 correspond to the functional groups present in bio composite sample. The IR peak observed ranged from 461.44 cm⁻¹ to 3981.50 cm⁻¹ and the percentage transmittance (%T) ranged between 10 and 100 % Focusing on the functional group region of the spectra i.e. wavelength ranging from 1500-4000 cm⁻¹. It can be observed that bio composite sample exhibited IR main peak positions of 1550 cm⁻¹, 2900 cm⁻¹, 3150 cm⁻¹, 3400 cm⁻¹, 3650 cm⁻¹ representing C=C, C-H, =C-H, N-H and O-H functional groups respectively.





3.7 Water absorption

Water absorption results showed that with increasing filler loading, particle size and immersion time (days), percentage water absorbed increased. Filler size 0.841mm with filler to matrix ratio 20% had the highest percentage water absorbed 0.93%, compared to 100% waste low density polyethylene which showed very little percentage water absorbed. Lignocellulosic fibers are known for their hydrophilic nature i.e they are permeable to water, significantly having effect on

water absorption, through hydrogen bonding free –OH (hydroxyl) groups come in contact with water which results in weight gain and water uptake in biocomposites (Zaaba *et al.*, 2013).

The Fourier transform infrared spectra of bio composite sample produced represented on Table 4 showed the presence of –OH (hydroxyl) functional group. Presence of cracks and voids were observed on the Scanning electron microscopy micrograph image referenced on Figure 11, cracks and voids could as well lead to the penetration of water molecules in the bio composite sample produced.



Figure 13: Percentage water absorbed against filler to matrix ratio and size

3.8 Impact test

Impact test assessed the response of produced bio composite samples when subjected to dynamic load, it showed the variation of impact strength to both filler size and filler to matrix ratio. It is clear from Figure 14 that with increasing filler loading impact strength decreases, deformability of waste low density polyethylene matrix occurred as a result of increased concentration of filler hence elasticity reduced.

Biocomposite samples with smaller particle size (0.42 mm) showed higher impact strength than bigger particle size (0.841 mm). Thus increasing the filler size with a higher filler to matrix ratio lead to a resultant decrease in impact strength, a probable reason can be placed to an increased level of stress concentration in the bio composite produced (Onuegbu and Igwe 2011).





3.9 Biodegradability test

Biodegradability test results shown on Figure 15, highlighted no observable weight change for 100% waste low density polyethylene, emphasizing the polymer matrix property of nonbiodegradability. Weight changes were observed for bio composite samples produced, it was observed that higher weight changes occurred for bigger particle size and filler loading to matrix ratios.



Figure 15: Weight reduced against filler to matrix ratio and size

4. Conclusion

Effects of selected factors (filler size and filler loading to matrix weight ratio) were investigated on produced bio composite samples where optimum value of tensile strength was obtained at 10% filler to matrix loading with particle size 0.42 mm which produced a Young's modulus of 320.321 N/mm². Higher particle size with higher filler loading elicited a higher value for compressive strength based on a force at peak value of 27552.500 N.

Flexural strength, impact strength and tensile strength all decreased with increasing filler loading. Percentage of water absorbed increased with increasing filler loading, particle size and immersion time. Biodegradability test showed higher weight changes for bigger particle size and filler loading to matrix ratios.

Application and suitability of *Parinari* waste low density polyethylene bio composite in the production of packaging materials and insulators, effects of chemical treatment on *Parinari* -waste low density polyethylene bio composite properties, determination of viscosity of molten waste low density polyethylene matrix and *Parinari polyandra* shell blend are recommended for further research.

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