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Sustainability of coconut shell residue as a filler with polystyrene on mechanical and morphology properties and elemental composition of the compisite

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ABSTRACT

This research is to develop environmentally friendly, lightweight composites using coconut shell, as filler in some thermoplastic polymer matrices Polystyrene (PS); to determine the mechanical properties of the coconut shell-residue polymer composite, to find if there is any new improvement over the properties of the starting thermoplastic polymer and determine the morphology and elemental composition of the composites. Coconut shell was collected from the surroundings of Ekwulumili in Nnewi-South L.G.A of Anambra State, Eastern Nigeria where they have been dumped after usage. The research was carried-out at JUNENG NIG LIMITED Enugu, Civil Engineering Department Laboratory University of Nigeria and Chemical Engineering Department Laboratory Ahmadu Bello University (ABU), Nigeria; between May 2016 and August 2018. The agro-wastes were grand into power and incorporated into the virgin thermoplastic polymers as filler at varied levels of 3%, 6%, 9%, 12% and 15%. The virgin HDPE thermoplastic polymers were used as the Control in the study. The mechanical properties of the composites produced were determined using American standard for Testing and Materials (ASTM), Standard Testing Methods; Scanning Electron Microscopy (SEM) was used to determine morphology while Energy Dispersive Spectroscopy (EDS) was used to determine the elemental composition of the composites. There was a significant improvement in tensile strength and tensile modulus of the blend coconut shell filler composites which were influenced by the amount of filler in the composites. The PS loaded with coconut shell filler has MoE (Modulus of Elasticity) at 3% of coconut shell filler has MoE of 893.22 MPa, 6% filler has MoE of 678.79 MPa, 9% filler has MoE of 1582.20 MPa, 12% filler has MoE of 1475.00 MPa and at 15 % filler has MoE of 780.12 MPa

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respectively. Then the value of MoE for pure PS matrix obtained in this work is 955.59 MPa. These results indicate that loading PS with 9% and 12% of coconut shell filler led to increase in the MoE which ultimately increased the brittle tendency of the polymer composites. Loading of PS with 6% and 15% of coconut shell filler respectively decreased the MoE of the polymer below the control sample suggesting that the ductility of the sample has been slightly increased. In tensile strengths of 13.98 MPa, 9.87 MPa, 41.94 MPa, 20.56 MPa, and 9.05 MPa were obtained for 3 %, 6 %, 9 %, 12 % and 15 % respectively while that of unloaded PS was 12.34 MPa. The results show that incorporation of coconut shell filler within the PS matrix improved in the tensile strength for composites formed with 3%, 9% and 12% of coconut shell filler while decrease in tensile strength were observed for composites formed with 6% and 15 % of coconut shell filler. Scanning electron microscopy (SEM) using imageJ software was carried out on the samples to estimate the average particle size of the polymer coconut shell waste. The micrograph reveals uniformity of smooth surface structure. This signifies homogeneous dispersion of the filler into the polymer matrix. It may also be because of low loading of the filler into the polymer matrix and also good adhesion between polymer matrix and dispersed phase. The elemental compositional analysis, using Energy Dispersive Spectroscopy (EDS) had all samples contain C and N as a major element present and others as trace; Fe, Zn, Ti, S, K, Al and Si. This study has provided different combinations of coconut shell-residue thermoplastic polymer composites which have potential application in the automobile and building construction industry. The utilization of agro-waste products in Nigeria and its degradation would help solve the problem of environmental pollution threat which they pose. Finally, the whole project would serve as a means of turning waste to wealth by utilizing agro-waste products in developing low cost polymer composites to serve a number of interesting applications

***Keywords*:** coconut shell, composites, PS matrices, polystyrene

1. INTRODUCTION

The abundant presence of natural fibre/filler and any other available agro-waste has also been responsible for latest development in research towards eco-friendly composite materials. In developed and under-developing countries especially in Nigeria, agro-waste products are produced in large quantities and cause environmental degradation, such as being burnt off or dumped in water bodies thereby causing environmental pollution.

The utilization of agro-waste products would help solve the problem of environmental pollution which they constitute. It will also serve as a means of turning waste to wealth by utilizing agro-waste products in developing a low-cost polymer composite to serve a number of interesting applications. Having discovered from previous works that particulate fillers or fibres are very good in reinforcing and enhancing the properties of polymer matrices, it is imperative to utilize waste from agricultural products and extracts obtained for material development and applications.

Composite is a material composed of two or more different components with the properties of the resultant material being superior to the properties of starting individual materials that make up the composite.

Therefore, from the definition of composite, blending or combining or fabricating of agro-waste and plastic (virgin or recycled) to obtain a material of superior properties to the single material for multi-functional applications could be composite. Studies on agro-waste and natural fibres composites have attracted due consideration from academicians and industrialists for their excellent properties such as improved mechanical strength, dimensional stability, wear resistance, low-cost, low-specific gravity, availability, and non-abrasiveness etc. In practical application, low-cost agro waste-fibre reinforced thermoplastic composites are gaining significant roles in building and automobile industries, and other consumer applications. More so, the inherent quality outputs of waste-plastic composite such as low cost, renewability, biodegradability, low specific gravity, availability, high strength and non-abrasiveness make the composites more suitable for use in a variety of practical applications. Underutilized agro-wastes are most importantly rich resources of lignocellulosic materials. Some typical examples are palm kernel shell, coconut shell, cow horn, millet, rice, wheat, corn straw, cocoa husk, corncobs and fibre.

Fillers not only reduce the cost of composites, but also frequently impart performance improvements that might not otherwise be achieved by the reinforcement and resin ingredients alone. Fillers are often referred to as extenders. In comparison to resins and reinforcements, fillers are the least expensive of the major ingredients. Fillers can improve mechanical properties as well as fire and smoke performance by reducing organic content in composite laminates.

Also, filled resins shrink less than unfilled resins, thereby improving the dimensional control of moulded parts. Important properties, including water resistance, weathering, surface smoothness, stiffness, dimensional stability and temperature resistance, can all be improved through the proper use of fillers.

The aim of this research is to develop environmentally friendly, lightweight composites using coconut shell, as filler in Polystyrene (PS) matrices; to determine the mechanical properties of the coconut shell-residue polymer composite, to find if there is any new improvement over the properties of the starting thermoplastic polymer and determine the morphology and elemental composition of the composites.

2. METHODOLOGY

Coconut shell was collected from the surroundings of Ekwulumili in Nnewi-South L.G.A of Anambra State, Eastern Nigeria where they have been dumped after usage. Commercial virgin High Density Polyethylene (HDPE) polymer matrices were purchased from one of the Petrochemicals Company, Nigeria. The equipment used were Monsanto Tensiometer, weighing balance, ventilated oven, 0.2 µm mechanical sieve, Scanning Electron Microscopy (Phenom, model proX SEM), Energy Dispersive Spectroscopy (EDS) and Universal Testing Machine (UTM) 5569A (JJ Lloyd, London, United Kingdom, capacity 1-20KN) in accordance with ASTM D638 for tensile strength. Zinc Stearate was used as a protective incorporated.

Coconut shell was washed with clean running water; sun dried and then was broken into pieces with mechanical grinding mill machine. The broken pieces were then ground produce fibre powder and then they were separated with 0.2 µm mechanical sieve to get the particle form.

Inside a beaker 1g NaOH was added into 99 ml of distilled water to make solution. After adequate drying of the fibres for 2 to 3 hours, the fibres were soaked in the prepared NaOH solution. The fibres were then taken for compression moulding and the particle sized of the filler used were 3g, 6g, 9g, 12g and 15g of coconut shell fillers.

The composites were prepared using the following blending formulation:

2. 1. Coconut Shell/Polymer Composite Formulation

Weight of Polymer matrices (g)	Weight of Agro-Wastes Filler in Composites (g)
100	0.0
97	3.0
94	6.0
91	9.0
88	12.0
85	15.0

One hundred grams (100g) each of polymer matrices were used as a starting material (Control) before reinforcement of various percentages such as 3%, 6%, 9%, 12% and 15% of egg shell fillers were added into the different polymer matrices used. Polymer matrices blended with particle size of the agro-wastes fillers were measured into a compression mould, for example 97g of HDPE matrix blended with 3g of coconut shell filler was measured before subjecting the mixtures to compression moulding to produce the composites. Zinc stearate was used as protective incorporated coated into polymer matrix composite to prevent adhesion to the plastic surface and it was mixed into resin for compression moulding. Polymer matrix composite was placed between them and then the mould was closed; heat and pressure were applied to obtain a homogeneous composite. A preheating time of about 1 hour at 120°C was needed for moulding and 30 minutes for cooling to get the solid moulding. Rapid cooling (quenching) was applied at the end of holding time. After processing, specimens were cut into the desired size and shape before the characterization of the samples. Each of the experiment was carried out severally in order to obtain accurate data.

2. 2. Mechanical Properties

All the tests were carried out using International Standards such as American Society for Testing Materials (ASTM) standards. Universal Testing Machine (UTM) 5569A was suitable for many mechanical tests of polymer matrix composites. The composites containing 3%, 6%, 9%, 12%, 15% w/w filler each were prepared and the mechanical properties examined. The parameters determined were tensile and modulus of elasticity.

2. 2. 1. Tensile strength and Modulus of Elasticity

Tensile strength test is a measurement of elasticity. This test was applied to observe the strength of the polymer matrix composites and it is common procedure for studying the stress–strain relationship. Flexural strength test is defined as the ability of materials to resist deformation under load or measurement of bending under pressure. This is used to measure the rigidity of the polymer matrix composites. A dog bone-shaped specimen was prepared according to International Standard (i.e. ASTM: D638) for tensile strength test; the equipment used was Tensometer and each of the property samples were tested several times.

Procedure:

i. The samples were cut into a dog bone-shaped specimen according to ASTM D638 (160 × 19 × 3.2) mm (Length × Breadth × Thickness).
ii. The chucks of the tensile test were fixed on the nose pieces of the tensometer.
iii. The test pieces were inserted one at a time into the tensile chucks and locked up appropriately.
iv. The tensometer graphs for each of parament at different level were fixed to the graph drum of the machine and ensured a firm grip.
v. The working fluid (mercury) of the machine and the load/ extension scale were properly set at zero.
vi. Gradual but continuous load through the longer handle of the machine was applied; this helped the working fluid to begin its movement.
vii. At each interval, the recording pin attached to the cursor was pressed down with the left hand while the right hand was gradually loading the machine.
viii. By so doing, the load / extension property of the test piece is drawn on the graph attached to the revolving recording drum.
ix. The test piece was removed when its failure brakes occurred, then the mercury level returns back through the varida glass tube to zero level.
x. The true values of the loads and extension were extracted and converted into stress/ strain.
xi. The stress / strain of the test pieces was calculated, using each of the values from the loads and extensions. Tensile strength and MoE of the test pieces were determined and measured after re-plotting the graph for Stress/ Strain.

Using ASTM D638 standard (160 ×19 × 3.2) mm, that is length = 160 mm, breadth = 19 mm and thickness = 3.2 mm.

$$\text{Stress} = \frac{P}{A_0}$$

where
P is the force,
A_0 is the cross-sectional area and unit is N/mm^2, 1 N/mm^2 = 1 MPa.

For cross-sectional Area, A_0 = breadth × thickness (depth)
A_0 = 19 × 3.2
A_0 = 60.8 0MPa

$$\text{Strain} = \frac{L_I - L_0}{L_0}$$

$$\Delta L = \frac{X}{L_0}$$

where
L_I = length after the test
L_O = initial length before the test (160 mm)

$$X = \frac{\text{Measured value}}{4}$$

Each value from extension is the measured value and 4 is the magnification of the test pieces drawn on the graph attached to the revolving recording drum.

The graph of the Stress / Strain of the test pieces were re-plotted to determine/ measure the Tensile strength and MoE of the test pieces. Tensile strength of each of the polymer matrix composite was calculated as maximum force divided by cross-sectional area

$$\text{Tensile strength} = \frac{P}{A_0}$$

where
P is the maximum force,
A_0 is the cross-sectional area.

2. 2. 2. Morphology and Elemental Composition Analyses

Morphology analysis using Scanning Electron Microscopy (Phenom, model proX SEM) served as an effective means for the investigation of morphology in the composite system; the Scanning Electron Microscopy (SEM) study of polymer-filler composite produced images of samples by scanning the surface with a focused beam of electrons.

Elemental Composition analysis using Energy Dispersive Spectroscopy (EDS) served as an effective means to discover the surface elemental composition and estimate their proportion at different position, consequently given an overall mapping of the sample.

3. RESULTS AND DISCUSSION

The coconut shell samples results generated at different percentage fillers of agro-wastes/polymer matrix composites were presented.

- **Tensile Strength Result for Modulus of Elasticity (MPa)**

Table 1. Modulus of Elasticity (MoE) values for coconut shell-waste/polymer matrix composite

			Different percentages fillers loading				
Agro-waste	**Polymer matrices**	**Control**	**3%**	**6%**	**9%**	**12%**	**15%**
Coconut shell	PS	955.59	893.22	678.79	1582.20	1475.00	780.12

Table on Modulus of Elasticity (MoE) values for coconut shelll-waste/polymer matrix composite at 3%, 6%, 9%, 12% and 15% agro-waste levels

❖ **Tensile Strength Test (MPa)**

Table on Tensile strength values for agro-waste/polymer matrix composite at 3%, 6%, 9%, 12% and 15% agro-waste levels

Table 2. Tensile Strength values for coconut shell-waste/polymer matrix composite.

			Different percentages fillers loading				
Agro-waste	**Polymer matrices**	**Control**	**3%**	**6%**	**9%**	**12%**	**15%**
Coconut shell	PS	12.34	13.98	9.87	41.94	20.56	9.05

Pictogram on MoE and Tensile Strength of Coconut Shell residue/PS Composite

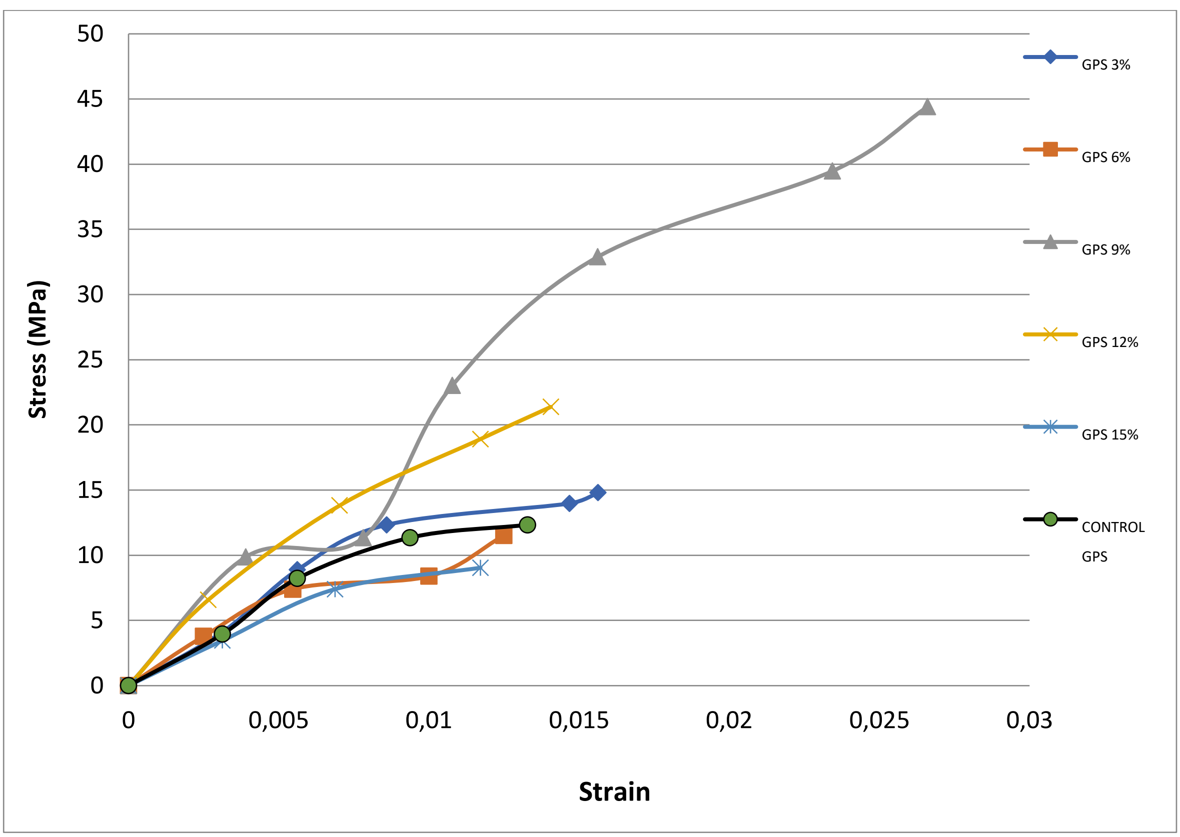


Fig. 1a. Stress-Strain Curves of the Control (PS) and PS- Coconut Shell composites at 3% - 15% Filler Levels

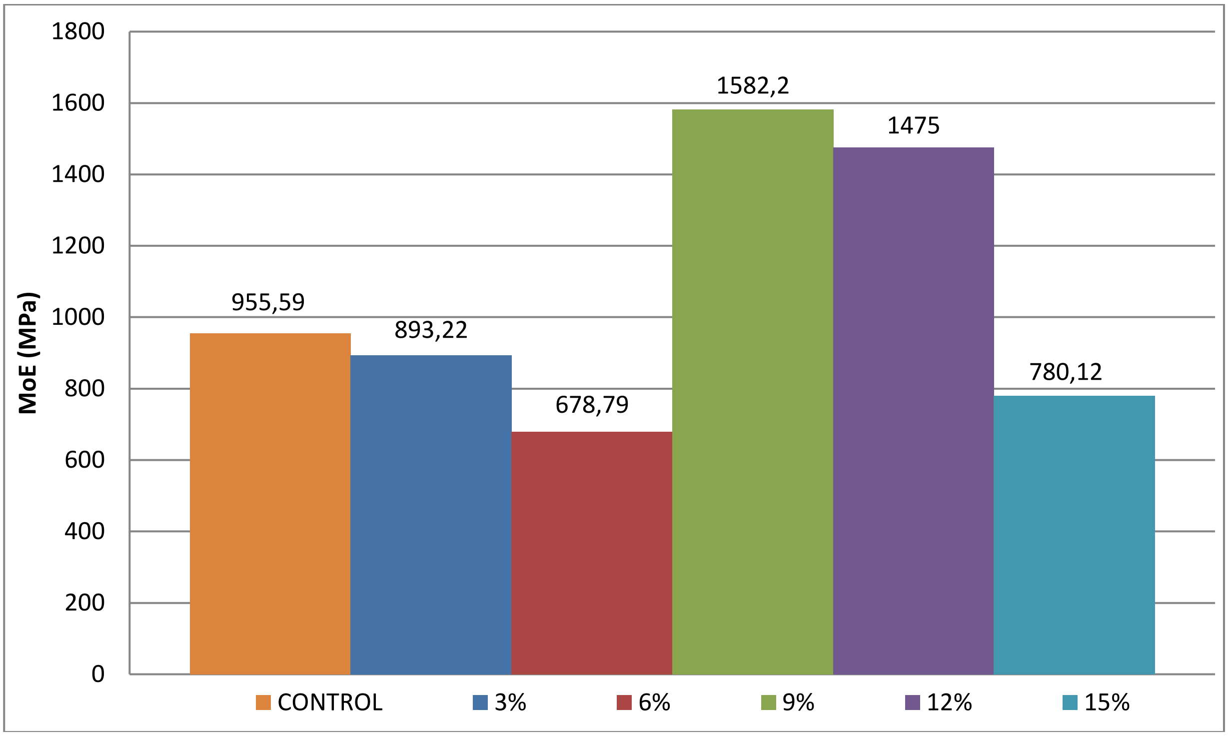


Fig. 1b. MoE Values of the Control (PS) and PS-Coconut Shell Composites at 3-15% Filler Levels

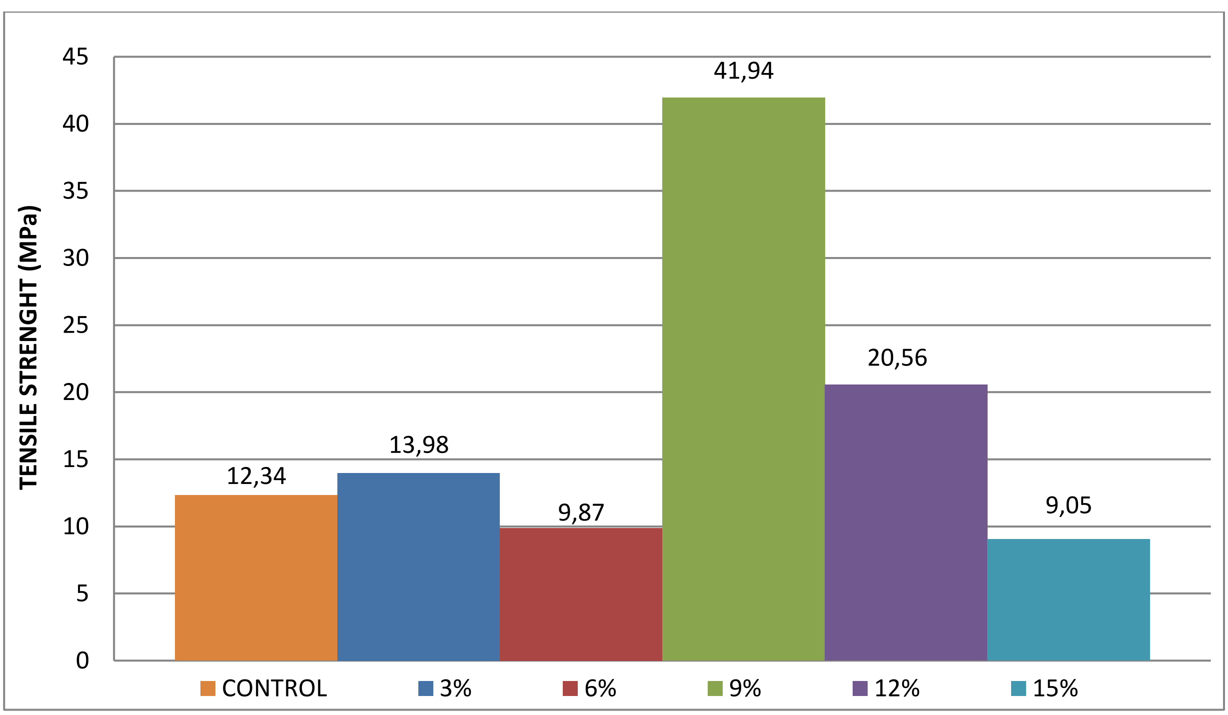


Fig. 1c. Tensile Strength Values of the Control (PS) and PS-Coconut Shell Composites at 3-15% Filler Levels

Surface Morphology of Agro-Wastes/Polymer Composites

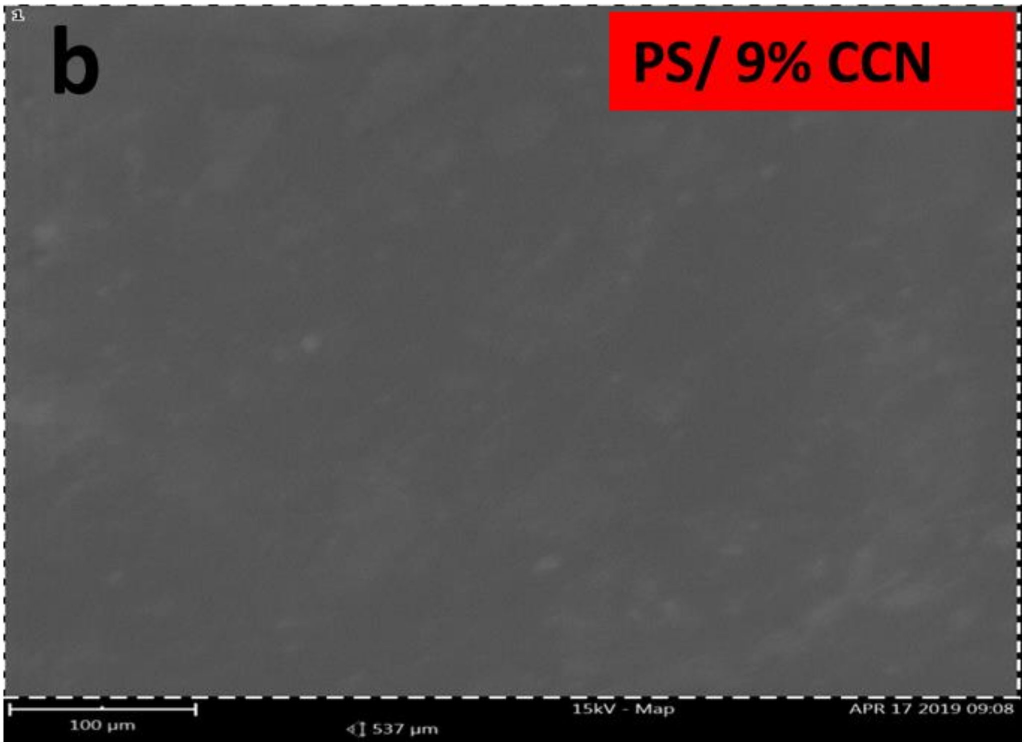
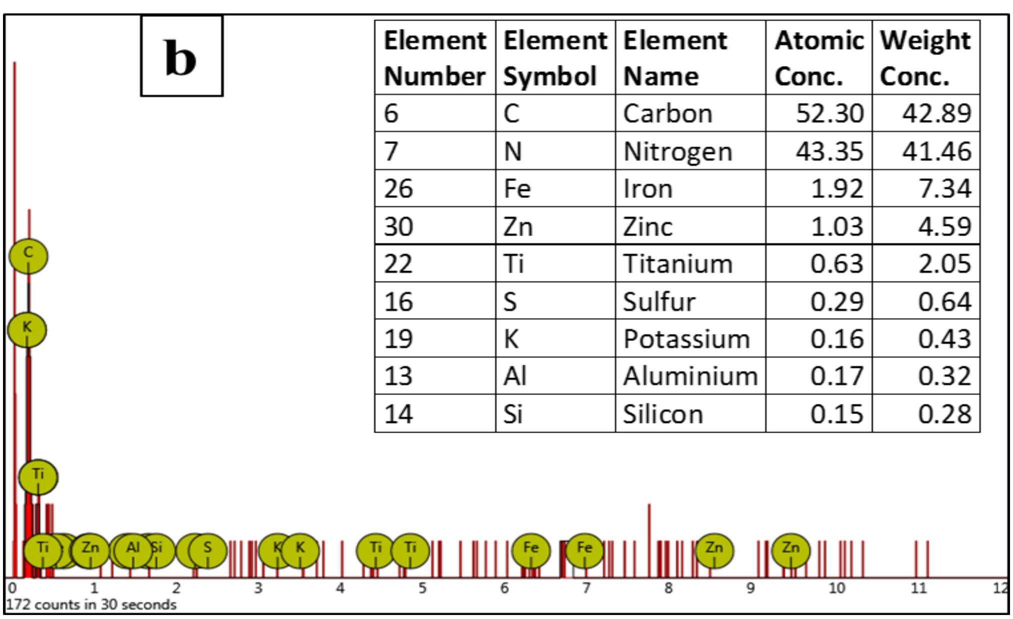


Figure 2. SEM Micrograph of PS loaded with 9% Coconut Shell Filler

Elemental Composition of Agro-Wastes/Polymer Composites



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	52.30	42.89
7	N	Nitrogen	43.35	41.46
26	Fe	Iron	1.92	7.34
30	Zn	Zinc	1.03	4.59
22	Ti	Titanium	0.63	2.05
16	S	Sulfur	0.29	0.64
19	K	Potassium	0.16	0.43
13	Al	Aluminium	0.17	0.32
14	Si	Silicon	0.15	0.28

Figure 3. EDS Spectra on Elemental Composition of PS loaded with 9% Coconut Shell

3. 1. Modulus of Elasticity (MoE)

The results of the modulus of Elasticity (MoE) and Tensile Strength tests on the composites of PS matrices reinforced with coconut shell filler respectively are presented in Tables 1-2 Figures 1(a-c) Figure 1a shows the plot of stress versus strain for different loads of coconut shell filler. The slope of the graph represents the modulus of elasticity (MoE) of the composite. Figure 1(b-c) shows the bar chart that depicts the variation of the MoE with percentage loading of coconut shell.

3. 1. 1. PS Polymer Matrix for MoE

PS polymer curve of stress – strain and PS loaded with 3%, 6%, 9%, 12% and 15% of coconut shell filler are also shown. The slopes of curves of PS give the MoE of the composites on the graphs, the PS loaded with 3% of coconut shell filler has MoE of 893.22 MPa, PS loaded with 6% filler has MoE of 678.79 MPa, PS loaded with 9% filler has MoE of 1582.20 MPa, PS loaded with 12% filler has MoE of 1475.00 MPa and PS loaded with 15 % filler has MoE of 780.12 MPa respectively. Then the value of MoE for pure PS matrix obtained in this work is 955.59 MPa. These results indicate that loading PS with 9% and 12% of coconut shell filler led to increase in the MoE which ultimately increased the brittle tendency of the polymer composites. Loading of PS with 6% and 15% of coconut shell filler respectively decreased the MoE of the polymer below the control sample suggesting that the ductility of the sample has been slightly increased.

3. 1. 2. PS Polymer Matrix for Tensile Strength

In PS, tensile strengths of 13.98 MPa, 9.87 MPa, 41.94 MPa, 20.56 MPa, and 9.05 MPa were obtained for 3 %, 6 %, 9 %, 12 % and 15 % respectively while that of unloaded PS was 12.34 MPa. The results show that incorporation of coconut shell filler within the PS matrix improved in the tensile strength for composites formed with 3%, 9% and 12% of coconut shell filler while decrease in tensile strength were observed for composites formed with 6% and 15 % of coconut shell filler.

3. 2. Surface Morphology of Polymer/Coconut Shell Composite

Figure 2 indicate the SEM images of PS coconut (CCN) composite and the estimated particle size of the micrograph PS/ 9% CCN obtained using ImageJ software was 29.73 μm. Micrograph of PS mixed with 9 % of CCN filler in which the composite reveals uniformity of smooth surface structure. This signifies homogeneous dispersion of the filler into the polymer matrix. It may also be because of low loading of the filler into the polymer matrix and also good adhesion between polymer matrix and dispersed phase.

3. 3. Elemental Composition of Polymer/Coconut Shell Composite

Figure 3 is the Energy Dispersive Spectroscopy (EDS) spectra and elemental composition of the PS/CCN composite. PS/CCN revealed the presence of carbon and nitrogen as the main elements; carbon has weight percentage value of 42.89 % corresponding to 52.30 atomic concentrations while nitrogen has percentage weight of 41.46 % which corresponds to 43.35 atomic concentrations. The high nitrogen concentration may be as a result of homogeneous dispersion of the filler material into the polymer matrix. Traces of other elements were observed in small amounts; the small quantities of these elements' present indicate that the agro-waste fillers are in small proportion compared to the polymer matrix. Trace of elements observed such as iron (Fe), zinc (Zn), titanium (Ti), sulfur (S), potassium (K), aluminium (Al) and silicon (Si) The presence of these elements (mostly the metals) in the prepared polymer is caused by the loading of the polymer with different amounts of coconut shell filler materials.

4. CONCLUSIONS

There was a significant improvement in tensile strength and tensile modulus of the blend coconut shell filler composites which were influenced by the amount of filler in the composites. The PS loaded with coconut shell filler has MoE (Modulus of Elasticity) at 3% of coconut shell filler has MoE of 893.22 MPa, 6% filler has MoE of 678.79 MPa, 9% filler has MoE of 1582.20 MPa, 12% filler has MoE of 1475.00 MPa and at 15 % filler has MoE of 780.12 MPa respectively. Then the value of MoE for pure PS matrix obtained in this work is 955.59 MPa. These results indicate that loading PS with 9% and 12% of coconut shell filler led to increase in the MoE which ultimately increased the brittle tendency of the polymer composites. Loading of PS with 6% and 15% of coconut shell filler respectively decreased the MoE of the polymer below the control sample suggesting that the ductility of the sample has been slightly increased. In tensile strengths of 13.98 MPa, 9.87 MPa, 41.94 MPa, 20.56 MPa, and 9.05 MPa were obtained for 3 %, 6 %, 9 %, 12 % and 15 % respectively while that of unloaded PS was 12.34 MPa. The results show that incorporation of coconut shell filler within the PS matrix improved in the tensile strength for composites formed with 3%, 9% and 12% of coconut shell filler while decrease in tensile strength were observed for composites formed with 6% and 15 % of coconut shell filler. Scanning electron microscopy (SEM) using imageJ software was carried out on the samples to estimate the average particle size of the polymer coconut shell waste.

The micrograph reveals uniformity of smooth surface structure. This signifies homogeneous dispersion of the filler into the polymer matrix. It may also be because of low loading of the filler into the polymer matrix and also good adhesion between polymer matrix and dispersed phase. The elemental compositional analysis, using Energy Dispersive Spectroscopy (EDS) had all samples contain C and N as a major element present and others as trace; Fe, Zn, Ti, S, K, Al and Si. This study has provided different combinations of coconut shell-residue thermoplastic polymer composites which have potential application in the automobile and building construction industry. The utilization of agro-waste products in Nigeria and its degradation would help solve the problem of environmental pollution threat which they pose. Finally, the whole project would serve as a means of turning waste to wealth by utilizing agro-waste products in developing low cost polymer composites to serve a number of interesting applications.

Author's contributions

- The study has provided several combinations of matrix/natural fillers that promote formation of new classes of composites and products with lower cost, light weight, high specific strength, flexibility, eco-friendly nature and availability.
- This research work has also shown that increased or decreased in the polymers, coupled with changes to other operating parameters, can have significant effects on plastic properties in modulus of elasticity and tensile.

- This study has provided different combinations of agro-waste/agro-residue thermoplastic polymer composites which have potential application in the automobile and building construction industry.
- The research has opened a new area of agro-wastes management for sustainable economy, creating job opportunities in industries and wealth creation.

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