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Mechanical Properties, Morphology and Elemental Composition of Composites Produced from Acrylonitrile-Butadiene-Styrene Thermoplastics Incorporated with Cow Horn

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ABSTRACT

The aim of this research is to develop environmentally friendly, lightweight composites using cow horn, as filler in Acrylonitrile-Butadiene-Styrene (ABS) polymers; to determine the tensile strength and tensile modulus properties of the cow horn-residue polymer composite, to find if there is any new improvement over the properties of the starting polymer and to determine the morphology and elemental composition of the composites. Cow horn was collected from the surroundings of Ekwulumili in Nnewi-South L.G.A and Nnewi-North both in 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 cow-horn were ground into powder and incorporated into the virgin thermoplastic polymers as filler at varied levels of 3%, 6%, 9%, 12% and 15%. The virgin thermoplastic polymers were used as the Control in the study. The tensile strength and modulus of elasticity property 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. The results generally showed significant improvements in tensile strength and tensile modulus of the blend ABS cow horn filler composites which were influenced by the amount of filler in the composites. ABS matrix loaded with 3% MoE of 516.72 MPa (Mega Pascal), ABS 6% MoE of 335.73 MPa, ABS matrix loaded with 9% of cow horn had MoE of 463.30 MPa, ABS matrix loaded with 12% of cow horn had MoE value of 209.24 MPa and ABS matrix loaded with 15% of cow horn had MoE of 236.36 MPa. MoE of 160.68 MPa for pure ABS matrix. The values obtained for tensile strength are 15.62 MPa, 9.05 MPa, 13.98 MPa, 10.69 MPa, 5.76 MPa and 6.58 MPa for 3%, 6%, 9%, 12%, 15% of cow horn filler and pure ABS matrix respectively. Scanning electron microscopy (SEM) was carried out on the samples using imageJ software to estimate the average particle size of the polymer cow horn. The micrograph reveals some agglomeration bulk and voids at the edges of the particles of the filler material with the polymer. The elemental compositional analysis, using Energy Dispersive Spectroscopy (EDS) has sample contains C and N as a major element present and others as trace; Fe, Na, Ag, Cu, Ti, Al, Mg, P, Zn, Si and S. This study has proven that combinations of lost-cost cow horn -residue filler reinforced with thermoplastic polymer composites are gaining significant roles in building and automobile industries, and other consumer applications. Also, 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: cow horn, ABS polymer matrix, composites, tensile, modulus of elasticity, morphology, elemental composition, Acrylonitrile-Butadiene-Styrene

1. INTRODUCTION

Composite be defined as a mixture or combination of two or more macro- or micro-constituents that differ in shape, chemical composition, and that are essentially insoluble in one another. Composite materials are materials produced from two or more constituent materials with notably dissimilar chemical or physical properties that, when merged, create a material with properties, unlike the individual elements. The trend in present days of environmentally friendly materials design and fabrication has triggered research into green composite due to challenges of global environmental concerns such as rising average global temperatures, rising sea level and decreasing polar ice cap etc [1-8, 10-16]. These have intensified pressures on researchers, academicians and industrialists towards manufacturing some new product designs using green materials partially or fully. The biodegradable waste disposal problem and benchmarks for cleaner as well as safer environment provide an abundant component of scientific research towards eco-composite materials. 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.

Cow horn is permanent pointed projection on the head of cow that consists of a covering of keratin and other proteins surrounding a core of live bone that grows out of the frontal bone of skull. It contains element such as calcium, phosphorus, magnesium and sulphur and trace element such as iron, zinc, manganese and cadmium [7, 9, 11].

The aim of this research is to develop environmentally friendly, lightweight composites using cow horn, as filler in some thermoplastic polymer matrix Acrylonitrile-Butadiene-Styrene (ABS); to determine the mechanical properties of the agro-residue polymer composite, to find

if there is any new improvement over the properties of the starting thermoplastic polymer and determine the morphology of the composites [17-26].

2. MATERIALS AND METHODS

2. 1. Sample Collection

Cow horn 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 Acrylonitrile-Butadiene-Styrene (ABS) polymer matrix was 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 1-20 KN) in accordance with ASTM D638 for tensile strength. Zinc Stearate was used as a protective incorporated.

2. 2. Pre-treatment of Sample

Cow horn 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 fillers 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:

Table 1. Cow Horn/Polymer Composite Formulation.

Weight of Polymer matrices (g)	Weight of Cow Horn 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 cow horn fillers were added into the different polymer matrices used. Polymer matrix blended

with particle size of the agro-wastes fillers were measured into a compression mould, for example 97g of ABS matrix blended with 3g of cow horn 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. 3. Mechanical Properties

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 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. 3. 1. Tensile Strength and Modulus of Elasticity Test

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. 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. While for flexural strength test; test piece was cut with respect to ASTM (300× 19 × 3.2) mm dimension.
- v. The chucks of the flexural tester were fixed on the nose piece of the tensometer.
- vi. The Sample were inserted into the 3-point flexural tester chamber and ensured a firm grip.
- vii. The tensometer graphs for each of paramant at different level were fixed to the graph drum of the machine and ensured a firm grip.
- viii. The working fluid (mercury) of the machine and the load/ extension scale were properly set at zero.
- ix. Gradual but continuous load through the longer handle of the machine was applied; this helped the working fluid to begin its movement.
- x. 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.

- xi. By so doing, the load / extension property of the test piece is drawn on the graph attached to the revolving recording drum.
- xii. The test piece was removed when its failure brakes occurred, then the mercury level returns back through the varida glass tube to zero level.
- xiii. The true values of the loads and extension were extracted and converted into stress/ strain.
- xiv. 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 , $1 \text{ N/mm}^2 = 1 \text{ MPa}$.

For cross-sectional Area, $A_0 = \text{breadth} \times \text{thickness (depth)}$

$$A_0 = 19 \times 3.2$$

$$A_0 = 60.80 \text{ MPa}$$

$$\text{Strain} = \frac{L_1 - L_0}{L_0}$$

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

where L_1 = length after the test

L_0 = initial length before the test (160 mm)

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

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

$$\text{Tensile strength} = \frac{P}{A_0} \quad \frac{X}{L_0}$$

where P is the maximum force,

A_0 is the cross-sectional area.

2. 3. 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 Cow horn samples results generated at different percentage fillers of agro-wastes/polymer matrix composites were presented.

❖ Modulus of Elasticity (MPa)

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

Table 2. Modulus of Elasticity (MoE) values for cow horn/polymer matrix composite

			Different percentages fillers loading				
Agro-Waste	Polymer matrices	Control	3%	6%	9%	12%	15%
Cow Horn	ABS	160.68	516.79	335.73	463.30	209.24	236.36

❖ Tensile Strength Test (MPa)

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

Table 3. Tensile Strength Test (MPa) values for cow horn/polymer matrix composite.

			Different percentages fillers loading				
Agro-Waste	Polymer matrices	Control	3%	6%	9%	12%	15%
Cow Horn	ABS	6.58	15.62	9.05	13.98	10.69	5.76

Pictogram on Tensile Modulus of Elasticity and Tensile Strength of Cow Horn/ABS Polymer

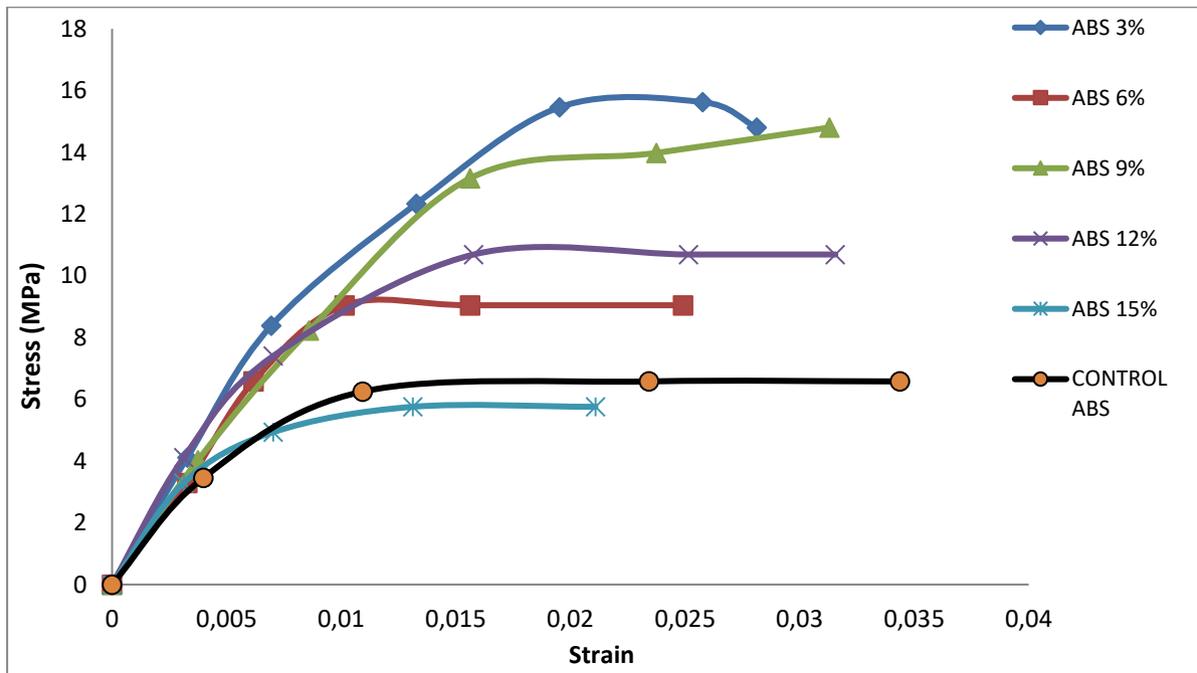


Fig. 1. Stress-Strain Curves of the Control (ABS) and ABS- Cow Horn composites at 3% - 15% Filler Levels

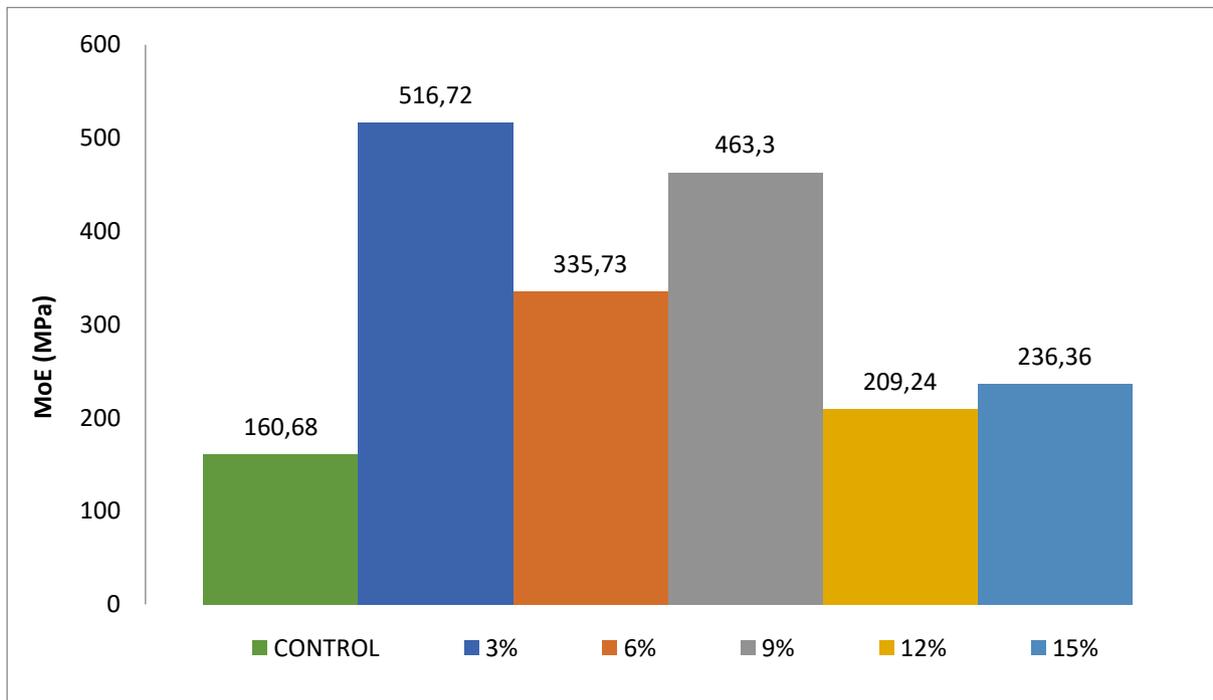


Fig. 2. MoE Values of the Control (ABS) and ABS-Cow Horn Composites at 3%-15% Filler Levels

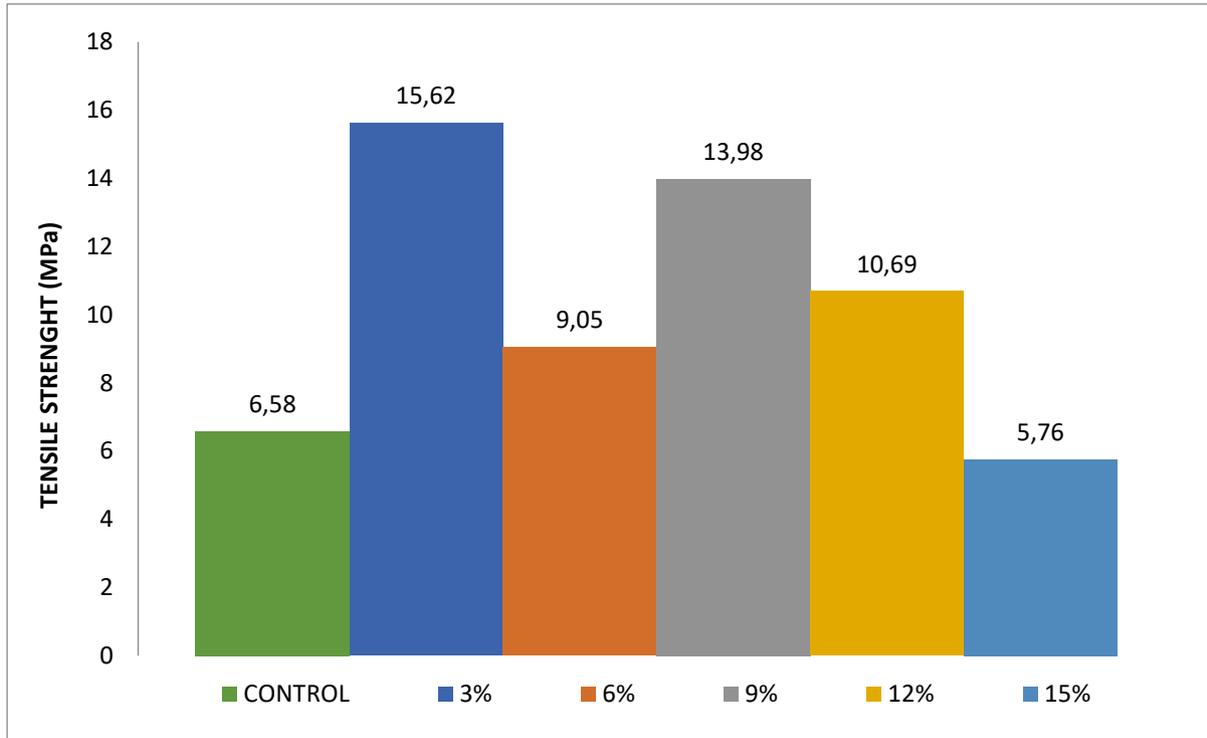


Fig 3. Tensile Strength Values of the Control (ABS) and ABS-Cow Horn Composites at 3%-15% Filler Levels

Surface Morphology of Cow Horn/Polymer Composites

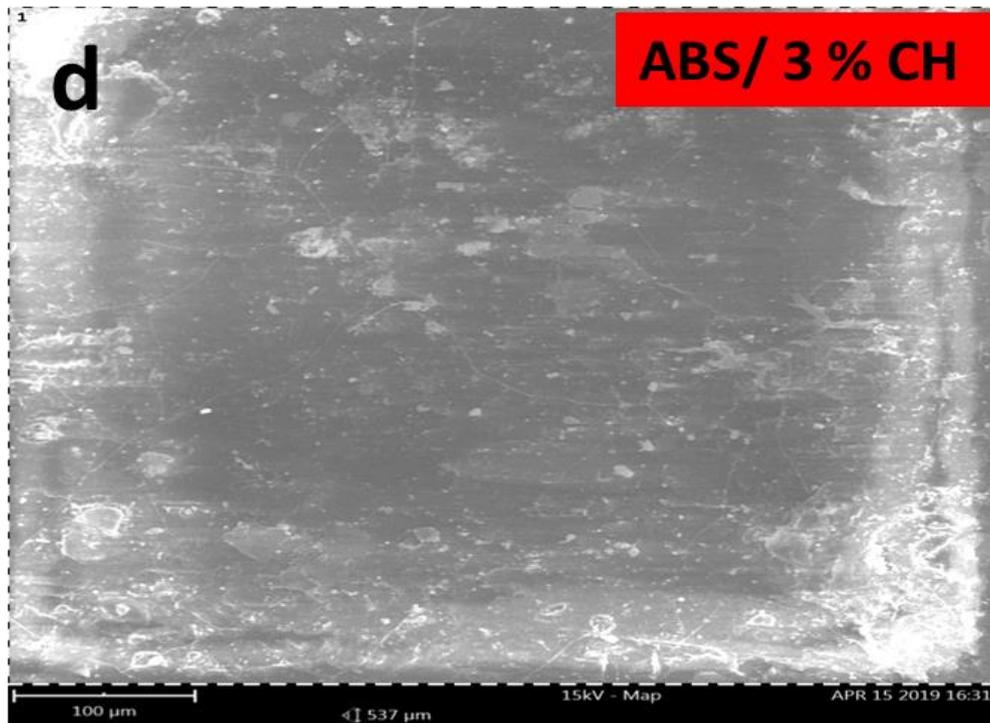


Figure 4. SEM Micrograph of ABS loaded with 3% Cow Horn Filler

Elemental Composition of Cow Horn/Polymer Composites.

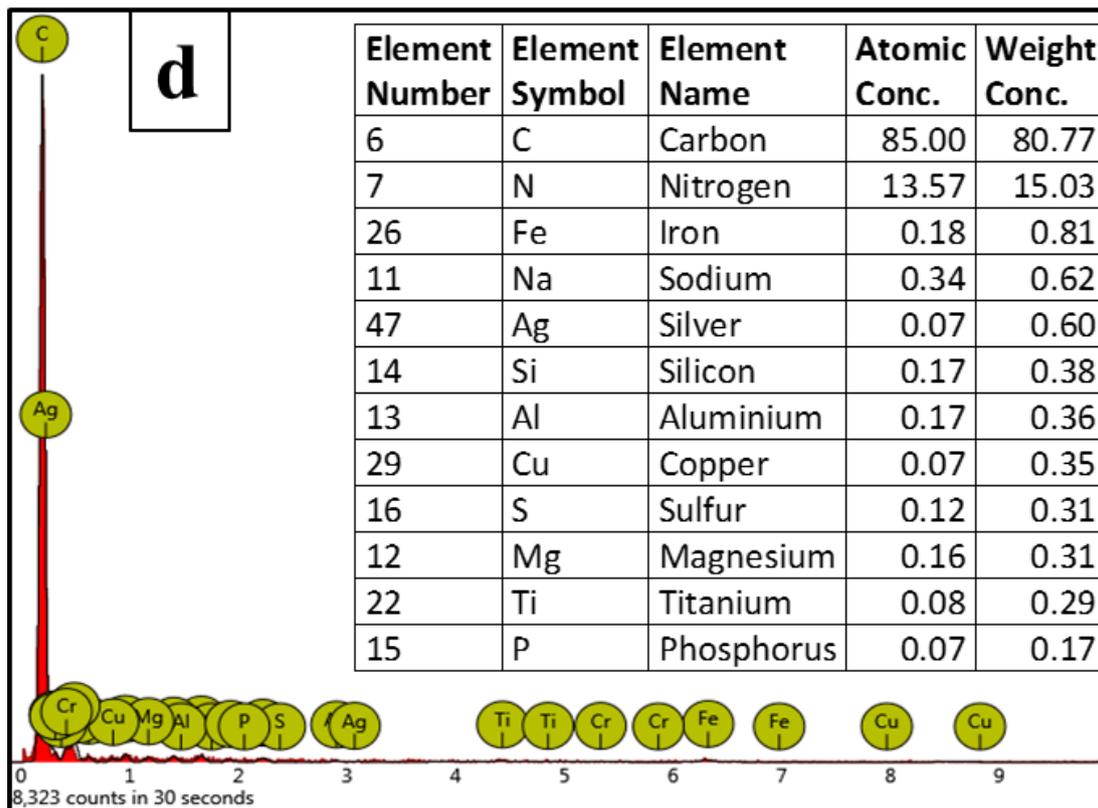


Figure 5. EDS Spectra on Elemental Composition of ABS loaded with 3% Cow Horn Filler

3. 1. Modulus of Elasticity and Tensile Strength

The modulus of elasticity (Young’s modulus) E is a material property that describes its stiffness and is therefore one of the most important properties of solid materials. The statistical correlation coefficient using Pearson product-moment between the control and cow horn filler loading on polymer matrices used at different percentages of stress and strain indicates a strong positive relationship between the variables. The correlations are statistically significant because their p -value is less than the significance level of 0.05.

The result of the cow horn filler loading reinforced on polymer matrices composites are shown in Tables 2 and Figures 1; the plot of stress – strain curves for ABS reinforced with different percentages of cow horn fillers are shown. The slopes of the graph represent the modulus of elasticity (MoE) of the composites and Figure 2 show the bar chart that represents the variation of the MoE with percentage loading of cow horn filler respectively. While Table 3 and Figure 3 show the tensile strength of ABS cow horn fillers composites.

3. 2. ABS Matrix for MoE

The stress – strain curves of ABS control matrix and its composites loaded with 3%, 6%, 9%, 12% and 15% of cow horn filler are shown in Figure 1. The slopes of the stress – strain

curve give the magnitude of the modulus of Elasticity (MoE) of the pure polymer and the composites in which there are variations in MoE due to addition of cow horn fillers. Figure 2 is the bar chart for comparison of MoE of ABS matrix with different percentages of cow horn fillers. Generally, an increase in MoE of the ABS matrix were observed due to incorporation of cow horn filler but this increase reduces in a zig-zig pattern as cow horn content increases in the polymer. ABS matrix loaded with 3% of cow horn had MoE of 516.72 MPa, ABS matrix loaded with 6% of cow horn had MoE of 335.73 MPa, ABS matrix loaded with 9% of cow horn had MoE of 463.30 MPa, ABS matrix loaded with 12% of cow horn had MoE value of 209.24 MPa and ABS matrix loaded with 15% of cow horn had MoE of 236.36 MPa. When compared with the MoE of 160.68 MPa for pure ABS matrix, it showed that cow horn filler had significant influence on the MoE of ABS matrix. The highest MoE of 516.72 MPa at 3% filler loading showed that there was homogeneous dispersion of the filler within the polymer matrix. But with increasing cow horn filler, the degree of homogeneous dispersion reduces leading to decrease in MoE.

3. 3. ABS Polymer Matrix for Tensile Strength

The chart for comparison of tensile strength of the polymer and its composites is shown for ABS on Table 2 and Figure 3. The tensile strength follows the same trend with the MoE of the ABS composites. The values obtained are 15.62 MPa, 9.05 MPa, 13.98 MPa, 10.69 MPa, 5.76 MPa and 6.58 MPa for 3%, 6%, 9%, 12%, 15% of cow horn filler and pure ABS matrix respectively. The result also depicts that there is more than 100% improvement in the tensile strength of ABS when 3% of cow horn filler was added while tensile strength of ABS when 15% of cow horn filler was added fall below the tensile strength of the pure ABS matrix. This suggests that at higher cow horn loading of 15%, the tensile strength of the composite is compromised.

3. 4. Surface Morphology of ABS Polymer/Cow Horn Composite

Scanning Electron Microscopy (SEM) serves as an effective means for the investigation of morphology of the composite system. According to (8), the SEM study of polymer filler composite helps in determining the distribution (dispersiveness) and compatibility between the filler and polymer matrix. In general, all the surfaces of the polymer composites are smooth with the particles appearing to be compact. In some of the composite structure, the particle of the filler material appeared to be homogeneously dispersed in the polymer matrix while some appeared to have formed an agglomerated mass of different dimensions. ImageJ software was used to estimate the average particle size of the polymer/agro – waste composite.

Figure 4 show the micrograph image of ABS prepared with loading of 3% cow horn (CH) filler. The estimated particle sizes from the micrographs of ABS/ 3% CH obtained using ImageJ software was 55.68 μm .

3. 5. ABS/Cow Horn Composite

Figure 4 shows the micrograph image of ABS/CH composite at 3 % filler loading. The micrograph reveals some agglomeration of the particles of the filler material with the polymer. This agglomeration is due to weak interaction between the particles of the filler and the polymer matrix. Presences of voids (white patches) along the edges show that the interfacial bonding between the particles of the filler and polymer matrix is weak.

3. 6. Elemental Composition of Polymer/Cow Horn Composite

Energy Dispersive Spectroscopy (EDS) relies on an interaction of some source of X-ray excitation on polymer filler composite. EDS helps to discover the surface elemental composition and estimate their proportion at different position, consequently giving an overall mapping of polymer filler composite. Figure 5 show the Energy Dispersive Spectroscopy (EDS) spectra and elemental composition of the ABS/CH composites.

3. 7. ABS/ Cow Horn Composite

Figure 5 shows that ABS/CH composite contains 80.77 % of carbon by weight which is equivalent to atomic concentration of 85.0 and 15.03 % of nitrogen by weight which is equivalent to 13.57 atomic concentrations. Atomic proportion of other trace elements is also given in Figure 5. The increase in atomic concentration of nitrogen could be attributed to the presence of nitrile group in chemical structure of ABS polymer matrix. Elemental composition of ABS/CH showed that they contain elements thus: carbon (C), nitrogen (N), iron (Fe), silicon (Si), aluminum (Al), magnesium (Mg), Sulfur (S) and phosphorus (P), sodium (Na), silver (Ag), zirconium (Zr), calcium (Ca), silicon (Si), zinc (Zn), titanium (Ti) and copper (Cu) at varying weight concentrations and their corresponding atomic concentrations. These elements observed are similar to elemental composition of cow horn obtained by (7) and (9). The presence of these elements (mostly the metals) in the fabricated polymer composites is because of the loading of the polymer with different amounts of cow horn filler materials.

5. CONCLUSION

There was a significant improvement in tensile strength and tensile modulus of the blend cow horn filler composites which were influenced by the amount of filler in the composites. ABS matrix loaded with 3% of cow horn had MoE (Modulus of Elasticity) of 516.72 MPa (Mega Pascal), ABS 6% of cow horn had MoE of 335.73 MPa, ABS matrix loaded with 9% of cow horn had MoE of 463.30 MPa, ABS matrix loaded with 12% of cow horn had MoE value of 209.24 MPa and ABS matrix loaded with 15% of cow horn had MoE of 236.36 MPa. When compared with the MoE of 160.68 MPa for pure ABS matrix, it showed that cow horn filler had significant influence on the MoE of ABS matrix.

The values obtained for tensile strength are 15.62 MPa, 9.05 MPa, 13.98 MPa, 10.69 MPa, 5.76 MPa and 6.58 MPa for 3%, 6%, 9%, 12%, 15% of cow horn filler and pure ABS matrix respectively. The result also depicts that there is more than 100% improvement in the tensile strength of ABS when 3% of cow horn filler was added while tensile strength of ABS when 15% of cow horn filler was added fall below the tensile strength of the pure ABS matrix. This suggests that at higher cow horn loading of 15%, the tensile strength of the composite is compromised. The micrograph image of ABS/CH composite at 3 % filler loading. The micrograph reveals some agglomeration of the particles of the filler material with the polymer. This agglomeration is due to weak interaction between the particles of the filler and the polymer matrix. Presences of voids (white patches) along the edges show that the interfacial bonding between the particles of the filler and polymer matrix is weak.

The elemental compositional analysis, using Energy Dispersive Spectroscopy (EDS) has sample contains C and N as a major element present and others as trace; Fe, Na, Ag, Cu, Ti, Al,

Mg, P, Zn, Si and S. This study has provided different combinations of cow horn/cow horn-residue thermoplastic polymer composites which have potential application in the automobile and building construction industry. The utilization of these 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. Also 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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