paddy, crop year, climate and geographical conditions (Chandrasekhar *et al.,* 2003).
Saw dust is a waste residue obtained from processing timber into various shapes and sizes (Obilade, 2014). Lack of proper storage can be a factor responsible for environmental pollution (Deac *et al.*, 2016). This fibres is usually used as sources of fuel by the villagers around the saw mill industry. The burning of the saw dust result into ash, called saw dust ash (SDA). The saw dust ash serve as an additional reinforcement to improve the properties of epoxy resin in this research work.

Epoxy is a thermoset formed by condensation polymerization reaction and during moulding acquire three dimensional network structure joined by strong covalent bonds. Thermosets are those which change irreversibly into hard and rigid materials on heating. After cooling, if the set article is again heated, it will not soften again, hence it is irreversible. Thermoset resins are usually, harder, stronger and more brittle than thermoplastic resins and they cannot be reclaimed from waste (Imanah *et al.,* 2015).

The aim of this research is to fabricate and characterize the rice husk ash/saw dust ash reinforced epoxy hybrid composite to see its applications in construction, automobile, aerospace, and plastic tank industries.
The objective was to determine the chemical composition of rice husk ash, saw dust ash using X-ray Florescence (XRF) and to investigate the impact, tensile, microstructure of fabricated RHA & SDA epoxy hybrid composite in order to see its safety in building construction, automobile, aerospace, plastic tank industries and also to provide a scientific method of disposing agricultural waste products.

## MATERIALS AND METHODS

### Materials

### Epoxy and Hardener

A commercially made epoxy resin (Grade 3554A) and the hardener (Grade 3554B) were purchased from Juneng Nigeria Limited in Nssuka LGA of Enugu State Nigeria and used as purchased.

### Rice Husk

Rice husk were obtain as a free gift from local rice farmer from Samaru, Zaria Kaduna State which burnt into ash, grinded and used to reinforce epoxy resin matrix in this research work.

### Saw Dust

Saw dust was collected from saw mill industry located in Hayin dogo Samaru-Zaria, which also burnt into ash as additional reinforcement for the epoxy resin matrix.

### Mould

The wood mould size 12mm x 10mm x 5mm was designed by a carpenter from Samara-Zaria before the rail-way station.

### Other material necessary

Container for mix the epoxy resin and hardener, stir rod, weigh balance, Sieve, and mould releasing agent (Mirror glaze).

## METHODS

### Rice husk preparation into ash

The rice husk were collected from local rice farmer in Samaru - Zaria Kaduna State Nigeria in large quantity and washed with water to remove foreign particles such as dust, small rice particles and fine sand particles. After thorough washing, it was dried at room temperature for 24 hours in order to remove moisture from it. The dried rice husk was burnt into ash by open burning in a metal container for 6 hours and left for 24 hours to cool down in Department of Building Ahmadu Bello University Zaria to

produce the rice husk ash used for this research work.

**Saw dust preparation into ash**

Sawdust was obtained from the local saw mills in Samaru - Zaria Kaduna State Nigeria. The sawdust was well prepared to remove the impurities. It was then dried for 24 hours at room temperature to release its moisture content. The dried sawdust was burnt into ash by open burning in a metal container for 6 hours to produce sawdust ash just like rice husk using the same method.

**The Formulation**

Figure: 1 **Process of burning rice husk and saw dust**

**Grinding**

The rice husk ash and sawdust ash obtained were grinded with grinded machine and later sieved with a sieve size 1.18mm.

**Mould Preparation**

A wood mould with dimension 12mm x 10mm x 5mm mould cavity was used in this research work. The mould cavity was well cleaned to remove any dust particle and then coated with a mould releasing agent (Mirror glaze) for easy removal of the laminate.

**Figure 2: Mould**

**Table 1: Weight Fraction Ratio of the Composite**

| Sample | Epoxy matrix | Rice Husk Ash | Saw Dust Ash |
|---|---|---|---|
| **1** | 100 | 0 | 0 |
| **2** | 90 | 7 | 3 |
| **3** | 80 | 7 | 13 |
| **4** | 70 | 7 | 27 |
| **5** | 60 | 7 | 33 |
| **6** | 50 | 7 | 43 |

**The Weight of the Composite Calculation**

$$W_{RHA} = \frac{WRHA}{(WRHA + WSDA + WM} \ x\ 100$$

$$W_{SDA} = \frac{WSDA}{(WRHA + WSDA + WM} \ x\ 100$$

$$W_{M} = \frac{WM}{(WRHA + WSDA + WM} \ x\ 100$$

$$W_{C} = W_{RHA} + W_{SDA} + W_{M}$$

Where;

$W_{RHA}$ = weight of the rice husk ash

$W_{SDA}$ = weight of the saw dust ash

Wc = weight of the composite

**Composite Preparation**

The rice husk ash, sawdust ash, epoxy resin and hardener were weighted at different proportion based on the total weight of the polymer matrix and fillers (Rice husk ash and sawdust) required for each sample with a weighing balance. Hand lay-up process was used for the composite production. For the control sample (1), epoxy resin and hardener were mixed in a ratio of 2:1 inside container without the fillers, stirred at low speed for 10 minutes and then poured into the prepared wood mould and allowed it to cure for 24 hours at room temperature. The composite sample (2) were made by mixing the measured volume of epoxy resin, hardener, pre-calculated weight of rice husk ash and sawdust ash based on this ratio (90:7:3) in a container and stirred with stirrer at low speed for 10minutes until the mixture became tacky and poured into the prepared wood mould and allowed to cure. The cured laminate was demoulded and further post-cured in an oven at 70ºC for 3 hours and then cut into different dimensions for characterization. The procedure was repeated for the sample 3, 4, 5, and 6 in the following ratio 80:7:13, 70:7:27, 60:7:33, and 50:7:43 respectively.

**Figure 3: The Composite**

## CHARACTERIZATION

### Chemical Compositions

The rice husk ash and saw dust ash obtained from the cooled container was packed and sieved to remove the broken net in the ash, grinded and sieved again with sieve size 4.75mm, then the ash was characterized using X-ray Florescence (XRF).

### Morphological characterization

### Scanning Electron Microscope (SEM)

The scanning electron microscope is one of the most versatile instruments used to study the morphology and chemical composition of materials when used in conjunction with energy dispersive X-ray spectrophotometry (EDX). It images the sample surface by scanning it with a high-energy beam of electrons. SEM can produce very high-resolution images of a sample surface, revealing details about less than 1 to 5 nm in size

### Mechanical Characterization

### Tensile Strength Test

Tensile strength or Ultimate tensile strength of a material can be determined by applying force to the test sample until it breaks. ASTM D638 is one of the most common plastic strength specifications and covers the tensile properties of unreinforced and reinforced plastics were used in this research. This test method uses standard “dumbbell” or “dog bone” shaped specimens. It is clamped at both ends and pulled at one of the clamped ends (usually downward) at constant elongation. The shape of the test specimen is designed to encourage failure at the thinner middle portion. The central section between clamps is called the initial gauge length, $L_0$. Due to the stretching of the specimen in tensile test, the initial test specimen length $L_0$ is increased to L and area $A_0$ is reduced to A. These test specimens, like all others, must be conditioned under standard conditions of humidity (50%) and temperature ($23^0$C) before testing.

### Impact Strength Test

Impact strength tests are measures of toughness or the ability of a specimen to withstand a sharp blow, such as being

dropped from a specific height or, as already noted, determined by the energy required to break a specimen. Izod impact test ASTM D256 was used in this work. In the Izod impact test (ASTM D256), the test piece is a cantilever, clamped upright in an anvil, with a V-notch at the level of the top of the clamp. The test piece is hit by a striker carried on a pendulum which is allowed to fall freely from a fixed height, to give a blow of 120ft-lb/in energy. After fracturing the test piece, the height to which the pendulum rises is recorded by a slave friction pointer mounted on the dial, The energy absorbed at fracture *E* can be obtained by simply calculating the difference in potential energy of the pendulum before and after the tests such as,

$$E = m.g\,(h-h')$$

Where *m* is the mass of pendulum and *g* is the gravitational acceleration.

## RESULTS AND DISCUSSION

### Results

**Table 2: The Chemical Composition of Rice Husk Ash and Saw dust Ash**

| Constituents | Mass Content (%) | |
|---|---|---|
| | Rice Husk Ash | Saw Dust Ash |
| $Na_2O$ | 0.089 | 0.645 |
| MgO | 2.727 | 2.867 |
| $Al_2O_3$ | 3.995 | 8.306 |
| $SiO_2$ | 74.097 | 55.871 |
| $P_2O_5$ | 0.883 | 3.307 |
| $SO_3$ | 0.794 | 2.947 |
| Cl | 0.029 | 0.720 |
| $K_2O$ | 3.373 | 1.421 |
| CaO | 1.590 | 8.002 |
| $TiO_2$ | 0.224 | 0.499 |
| $Cr_2O_3$ | 0.002 | 0.006 |
| $Mn_2O_3$ | 0.347 | 0.261 |
| $Fe_2O_3$ | 11.786 | 14.710 |
| ZnO | 0.057 | 0.087 |
| CrO | 0.007 | 0.351 |
| **Total:** $SiO_2+Al_2O_3+Fe_2O_3$ | 89.878 | 78.887 |

**Scanning Electron Microscope**

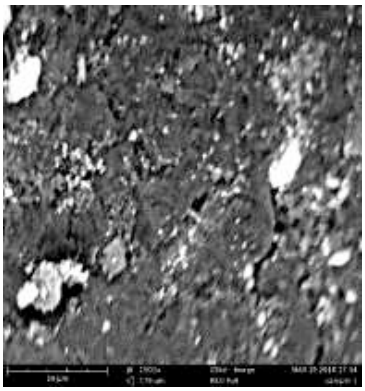

**Figure 4:** Sample 1at 1000 X mag.

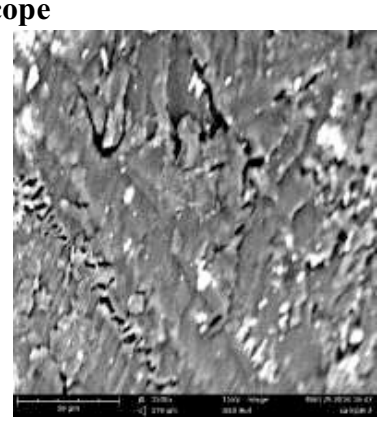

**Figure 5:** Sample 2 at 1000 X mag.

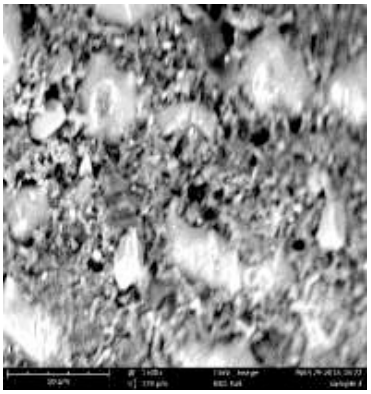

**Figure 6:** Sample 3at 1000 X mag.

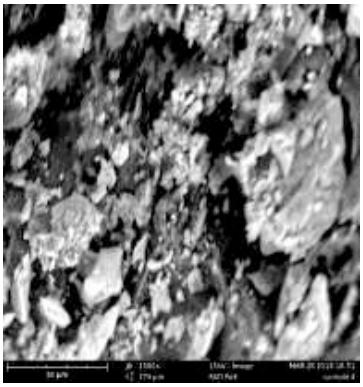

**Figure 7:** Sample 4 at 1000 X mag.

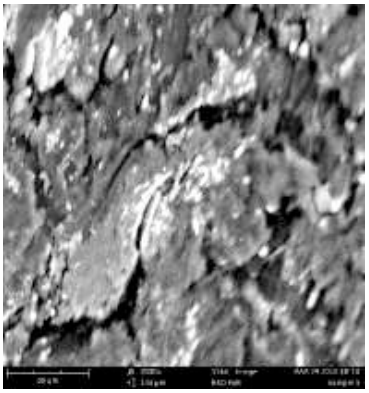

**Figure 8:** Sample 5at 1000 X mag.

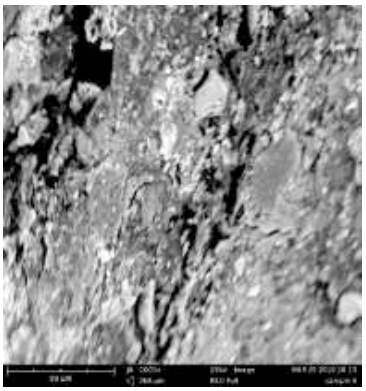

**Figure 9:** Sample 6 at 1000 X mag.

**Table 3: Tensile strength test results**

| Sample | Thickness (mm) | Width (mm) | Breaking load (mm) | Gauge length (mm) | Area ($mm^2$) | Average Area ($mm^2$) | Average Breaking load (mm) | Average Tensile Strength ($N/mm^2$) |
|---|---|---|---|---|---|---|---|---|
| 1 | 5.0 | 9.7 | 636 | | 48.50 | | | |
| | 5.0 | 10.0 | 510 | 40 | 50.00 | 49.00 | 542 | 11.06 |
| | 5.0 | 9.7 | 480 | | 48.50 | | | |
| 2 | 4.8 | 10.2 | 402 | | 53.76 | | | |
| | 5.0 | 10.8 | 456 | 40 | 54.00 | 51.44 | 432 | 8.40 |
| | 4.8 | 9.7 | 438 | | 46.56 | | | |
| 3 | 5.0 | 10.0 | 384 | | 55.00 | | | |
| | 5.0 | 10.5 | 378 | 40 | 52.50 | 53.15 | 364 | 6.85 |
| | 4.9 | 9.6 | 330 | | 51.94 | | | |
| 4 | 5.0 | 10.3 | 552 | | 51.50 | | | |
| | 5.0 | 9.3 | 384 | 40 | 46.50 | 48.33 | 444 | 9.19 |
| | 5.0 | 9.4 | 396 | | 47.00 | | | |
| 5 | 5.0 | 10.8 | 636 | | 54.00 | | | |
| | 5.0 | 10.6 | 450 | 40 | 53.00 | 52.67 | 540 | 10.25 |
| | 5.0 | 10.2 | 534 | | 51.00 | | | |
| 6 | 4.9 | 10.5 | 468 | | 51.45 | | | |
| | 5.0 | 10.2 | 576 | 40 | 51.00 | 51.98 | 504 | 9.70 |
| | 5.0 | 10.7 | 468 | | 53.50 | | | |

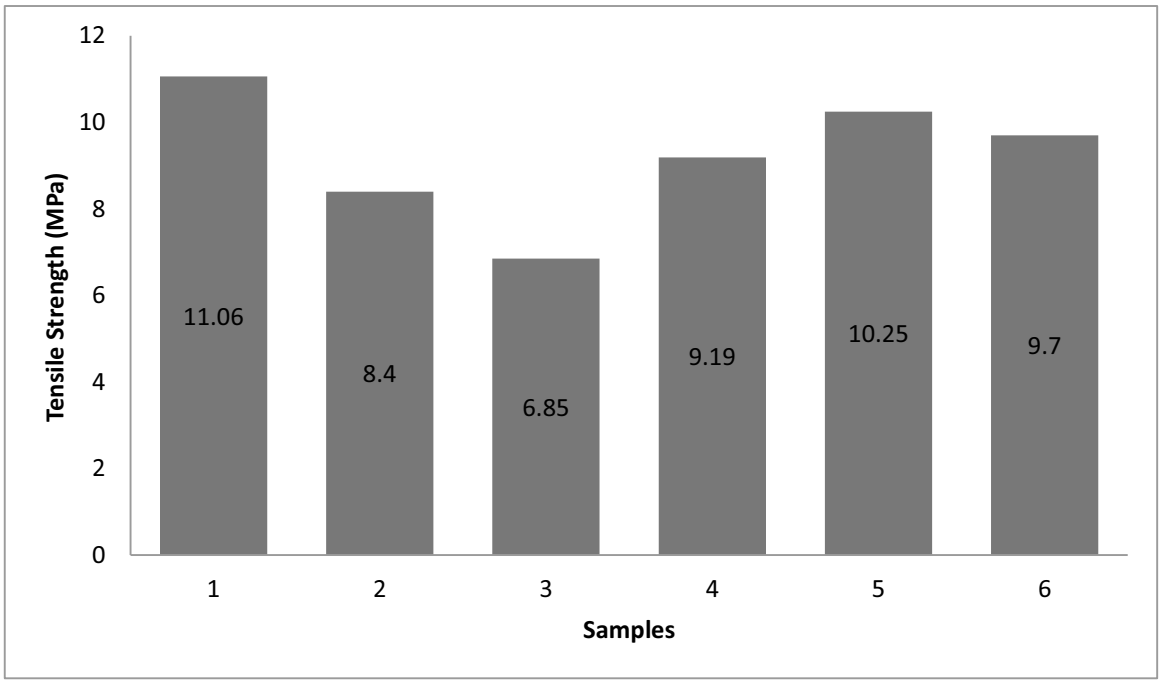


**Figure 10:** Tensile Strength of RHA & SDA Epoxy Hybrid Composite.

**Table 4: Impact Test results**

| Sample | Width (mm) | Thickness (mm) | Area (mm) | Impact energy (KJ) | Impact strength ($J/mm^2$) |
|---|---|---|---|---|---|
| **1** | 10.0 | 5.0 | 50.0 | 0.130 | |
| | 10.0 | 5.0 | 50.0 | 0.407 | 4.44 |
| | 10.0 | 4.9 | 49.0 | 0.122 | |
| **2** | 10.5 | 5.0 | 52.5 | 0.158 | |
| | 9.3 | 5.0 | 46.5 | 0.120 | 4.69 |
| | 9.9 | 5.0 | 49.5 | 0.436 | |
| **3** | 10.0 | 4.8 | 48.0 | 0.271 | |
| | 10.6 | 4.8 | 50.9 | 0.243 | 4.70 |
| | 10.0 | 5.0 | 50.0 | 0.186 | |
| **4** | 10.5 | 5.0 | 52.5 | 0.273 | |
| | 9.7 | 5.0 | 48.5 | 0.266 | 4.98 |
| | 9.8 | 5.0 | 49.0 | 0.208 | |
| **5** | 10.2 | 5.0 | 51.0 | 0.191 | |
| | 10.0 | 4.9 | 49.0 | 0.269 | 5.08 |
| | 9.8 | 5.0 | 49.0 | 0.296 | |
| **6** | 10.8 | 5.0 | 54.0 | 0.257 | |
| | 9.7 | 5.0 | 48.5 | 0.162 | 5.65 |
| | 10.0 | 5.0 | 50.0 | 0.422 | |

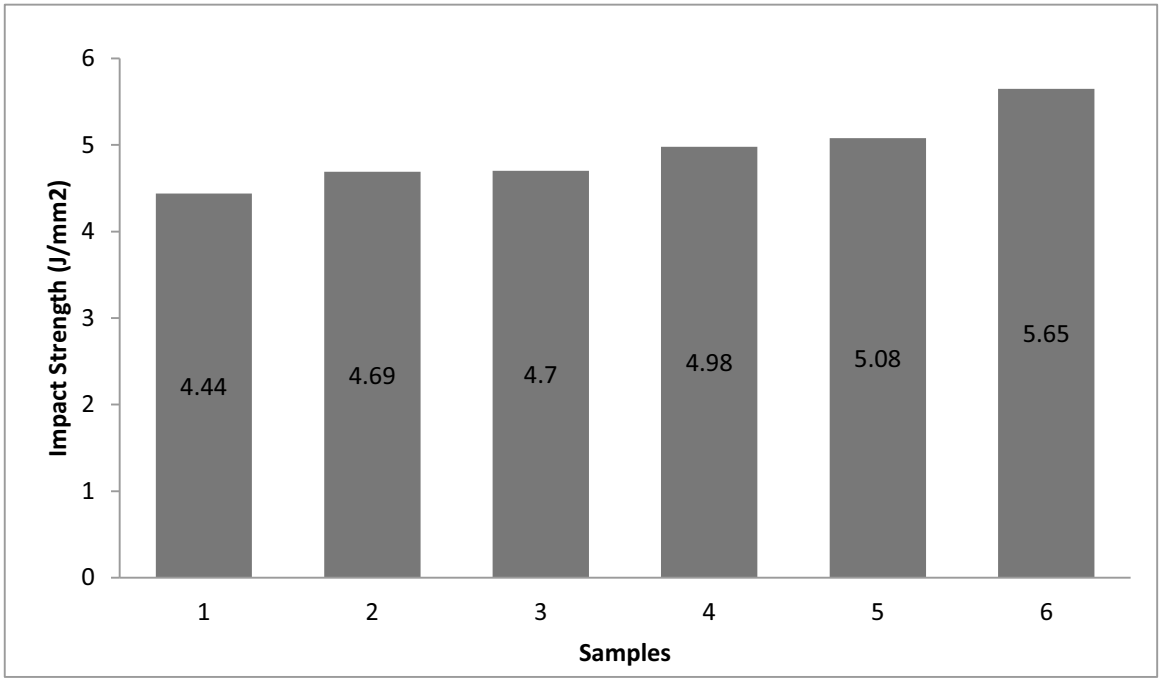


**Figure 11:** lmpact Strength of RHA & SDA Epoxy Hybrid Composite.

## DISCUSSION OF EXPERIMENTAL RESULTS

### Chemical Compositions

The Table 2 summarized the chemical composition of rice husk ash and sawdust ash together. In the table, $SiO_2$ amount to 74.097Wt %, while $Al_2O_3$ have 3.995Wt % and $Fe_2O_3$ is equal to 11.786Wt % in RHA column. In SDA column, $SiO_2$ amount to 55.871Wt %, while $Al_2O_3$ have 8.306Wt % and $Fe_2O_3$ is 14.710Wt %. The combination of ($SiO_2$ + $AlO_3$ + $Fe_2O_3$) in RHA column is 89.878Wt % which is 2.368Wt % less than 87.51Wt % (Swaminathen, *et al.,* 2016) and 4.682Wt % more compare to 94.56Wt % (Foong *et al.,* 2015). The combination of ($SiO_2$ + $AlO_3$ + $Fe_2O_3$) also in SDA column is 78.887Wt % which is 3.997Wt % more than 74.89Wt % averages of three samples in Raheem *et al.,* 2013. The total of the combination in both side are more than 70% as stipulated in ASTM C618. This confirm that rice husk ash and saw dust ash has a pozzolanic material and is capable of improving the properties of a composite.

### Scanning Electron Microscope (SEM)

Figure 4 showed the micrograph at 1000 X magnification for Sample (1) of ratio 100% epoxy matrix, 0% RHA, and 0% SDA. There were voids on the surface of the laminate, poor cohesion in some areas and fairly good cohesion is observed in some regions due to the mixing between epoxy resin and hardener.

Figure 5 produced SEM image at 1500 X magnification for Sample (2) of ratio 90% epoxy matrix, 7% RHA, and 3% SDA. Porosity was observed which may be due to gases and volatiles from epoxy matrix. Crack propagation occurred in the sample, which

may be attributed to curing process and thermal shrinkage during fabrication.

Figure 6 showed the microscopic structure at 1500 X magnification for Sample (3) of ratio 80% epoxy matrix, 7% RHA, and 13% SDA. There is zone of high concentration of epoxy matrix and fillers, which may be due to increase in proportion in the formulation, also crack propagation due to two different zones observed in the sample.

Figure 7 showed the micrograph at 1500 X Magnification of sample (4) of ratio 70% epoxy matrix, 7% RHA and 27% SDA. Epoxy matrix debonding and fillers pulled out observed within the laminate fabricated in some region due to cohesion between the epoxy matrix and the fillers or environmental attack. In another region, crack propagation was observed too due to the separation that occurred between the epoxy matrix and fillers.

Figure 8 produced the microscopic structure at 2000 X magnification for sample (5) of ratio 60% epoxy matrix, 7% RHA, and 33% SDA. There is debonding in some region which latter lead to delamination due to separation of the layers of reinforcement or poor processing during production.

Figure 9 showed the micrograph at 1000 X Magnification of sample (6) of ratio 50% epoxy matrix, 7% RHA and 43% SDA. Good cohesion was observed at the interface between the epoxy matrix and the filler due to interaction between the adhesive and reinforcement, debonding occurred which may be due to cohesive attraction between the resin and reinforcement agents and voids was also observed which may be due to contamination or mechanical fault during processing.

**Tensile Strength**

Figure 10 showed the tensile strength of RHA & SDA epoxy hybrid composite. In the figure, the tensile strength 11.06MPa, 8.40MPa and 6.85MPa of Sample 1 to 3 respectively decrease gradually which may be due to level of dispersion of RHA and SDA in the epoxy matrix but sharp increase in tensile strength 9.19MPa, 10.25MPa in sample 4 and 5 respectively was observed, which may be attributed to strong degree of adhesion between the RHA and SDA with the epoxy matrix and tensile strength of 9.70MPa was also observed in sample 6 which may be due to weak of adhesion between the RHA and SDA with the epoxy matrix. The overall Ultimate tensile strength for RHA/SDA epoxy hybrid composite is 9.19MPa when compare to 8.93MPa (Antaryami, 2017) which is the highest ultimate tensile strength in his research work, followed by strain of 0.38m/m less than the maximum and minimum strain value of 19.10m/m and 2.69m/m respectively (Rahul Kumar, *et al.,* 2014) and Young's modulus of 24.18GPa

**Impact Strength**

Figure 11 showed the impact strength of RHA & SDA epoxy hybrid composite. In the figure above, the impact strength; 4.44J/mm$^2$, 4.69 J/mm$^2$, 4.70 J/mm$^2$, 4.98 J/mm$^2$, 5.08 J/mm$^2$, and 5.65 J/mm$^2$ increases respectively as the mass of fillers in each sample increases, which may be due to wettability interaction between the epoxy matrix and fillers in the composite.

**CONCLUSION**

From the analysis carried out, the following conclusions can be drawn; sample 6 with 50% epoxy matrix, 7RHA and 43% SDA have excellent mechanical strength and from its morphological characterization point of view, there is good cohesion between epoxy matrix and fillers. Therefore it will be suitable in automobile industry to produce interior and exterior auto parts. Also in aerospace industry to produce window frames, breaking system due to its

lightweight. Sample 2 with 90% epoxy matrix, 7% RHA and 3% SDA will not be suitable due to its porosity and some crack observed in its microstructure. Sample 5 with 60% epoxy matrix, 7% RHA, and 33%SDA can be recommended in construction and plastic tank industries due to its strong mechanical properties. Sample 1 with 100% epoxy matrix, 0% RHA, and 0% SDA will not be suitable because as filler increases, mechanical properties also increase.

**ACKNOWLEDMENT**

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**RECOMMENDATIONS**

The authors recommended addition of colorant to the formulation in order to make the composite looks attractive when producing wall tiles for structural application. Furthermore, other mechanical test, thermal analysis should be carried out on rice husk ash & sawdust ash reinforced epoxy hybrid composite.

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# Mechanical and Viscoelastic Properties of Plantain Peel Powder Reinforced Recycled High Density Polyethylene Composites

**Jacob J.[1*], Mamza P.A.P[1], Ahmed A.S[2] , and Yaro S.A[3]**

[1]Department of Chemistry, Ahmadu Bello University, Zaria
[2]Department of Chemical Engineering, Ahmadu Bello University, Zaria
[3]Department of Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria

## ABSTRACT

The influence of alkaline treated plantain peel powder incorporation on the mechanical and viscoelastic properties of recycled high density polyethylene (RHDPE) was investigated. Composites samples of varying weight fraction of reinforcement: 5%, 10%, 15%, 20% and 25% were prepared via melt mixing and compression moulding techniques. Dynamic mechanical properties were evaluated using 242 E dynamic mechanical analyzer in a temperature range from 30-100$^0$C at a frequency of 1 Hz. Tensile strength, elastic modulus and hardness test data showed an improvement in mechanical properties which were found to increase with increase in weight fraction of reinforcement. Similarly, dynamic mechanical properties results revealed an increase in storage modulus (E') with increase in weight fraction of reinforcement and decrease with increase in temperature. Optimum E' value of 1007.4 MPa was obtained at 25%, with lower values of loss modulus and damping. The results indicated an improvement in the viscoelastic properties and load bearing capacity of RHDPE with plantain peel powder addition. Scanning Electron Micrographs showed uniform distribution of plantain peel powder in the RHDPE matrix which explained the observed improvement in thermo-mechanical properties.

**Keywords:** *Composites, Dynamic Mechanical Analysis, Mechanical properties, Plantain peel powder, Recycled polyethylene*.

## 1. INTRODUCTION

Polyethylene (PE) is one of the most widely used thermoplastic in the world because of its good properties such as toughness, near-zero moisture absorption, excellent chemical inertness, low coefficient of friction and ease of processing (Khanam and Al Maadeed, 2015).The precise degree of crystallinity and density are dependent on the molecular weight of PE (Usman *et al.,* 2016*).* Generally, the most used PE grades are low density polyethylene (LDPE), medium density polyethylene (MDPE) and high density polyethylene (HDPE). HDPE in particular exhibits excellent performances such as chemical stability barrier, mechanical strength, and dielectric properties (Musa *et al*., 2017), and is the third-most frequently used plastic in the world (Kumar *et al.,* 2011). These properties makes HDPE a versatile material in the manufacture of many products and in packaging such as milk jugs, detergent bottles, margarine tubs, garbage containers (Klyosov, 2007); water pipes (Vasile and Pascu, 2005) and bottle caps resulting in a large volume of wastes. Above all, HDPE has a strong plasticity and deformability (Pawlak and Galeski, 2005), being widely used in the preparation of polymer composites (Zhang *et al*., 2017).

Plastic waste recycling in form of composites is a preferable way of addressing this environmental challenge.

* *Corresponding author: jacobumar@gmail.com +2348064201647*

Over the last few years, different types of waste materials (plastic waste and natural fibres) have been utilized in composite production; which offers an opportunity for waste material re-utilization and thereby reduce environmental pollution (Eze *et al*., 2013). Availability, ease of processing, environmental friendliness and biodegradability are some of the properties that made natural fibres to have an edge over synthetic fibres like glass and Kevlar in composite manufacture.

Several natural fibres have been investigated in this regard including but not limited to banana fibre (Naidu *et al*., 2013), rice husk (Tong *et al.*, 2014), pineapple peel (Danladi and Shui'ab, 2014), bagasse (Naguib *et al.,* 2015), kenaf core powder (Majid *et al*., 2016) and jute (Gupta, 2018). However, despite the properties and applications of natural fibre-based composites their hydrophilic property poses a serious challenge in their effective use as they are not well compatible with polymer matrix which is hydrophobic (Nguong *et al.,* 2013; Shehu *et al.,* 2017).

To overcome this therefore, the fibre surface has to be modified (Hossain *et al*., 2014; Fiore *et al*., 2015; Mohanta, 2016). The modification could be physical or chemical and its of various types. Chemical treatment process removes non cellulosic substance such as impurities, waxes, pectin, hemicelluloses, lignin which covers the cellulose fibrils and binds these fibrils together (Usman *et al*., 2016).

Removal of these substances also gives rise to a rougher fibre surface resulting in an increase in surface contact of the fibre and the matrix. This causes an improvement of mechanical interlocking between the polymer and the fibre, leading to enhanced mechanical properties such as tensile strength, flexural strength, hardness index and impact strength. Equally important is the need to evaluate the dynamic mechanical properties of PE composites in order to establish its applications like casing of electronic instruments such as mobiles, laptops and so on (Gupta, 2018).

Dynamic mechanical analysis (DMA) is a technique that yields information about the viscoelastic behaviour, dynamic fragility, storage modulus, loss modulus, stress relaxation modulus, effective constant of reinforcement, creep compliance and damping parameter as a function of frequency, temperature, time, atmosphere or combination of these parameters (Gupta and Bharti, 2017). Some researchers have investigated the dynamic mechanical analysis of natural fibre based composites and reported that these materials have optimum properties suitable for different applications. Dan asabe (2016) studied the thermo-mechanical characterization of banana particulate reinforced PVC composite as piping material. Through dynamic mechanical analysis, the composition with optimum mechanical property of 42MPa was estimated to have a long term stress value of 25MPa corresponding to 40% loss in strength over a period of 32 years. The effect of variation in frequencies on dynamic mechanical properties of jute fibre reinforced epoxy composites was reported by Gupta (2018). The results indicated that thermal stability and load bearing capacity was found to improve with increase in fibre loading but decrease with increase in temperature. This current research therefore seeks to determine the mechanical and viscoelastic properties of plantain peel reinforced RHDPE composites with a view in determining its suitability for different applications.

Table 1: The designation of symbol and their definition as used in this work

| Symbol | Definition |
|---|---|
| A0 | unreinforced RHDPE (control) |
| A5 | 5 wt% plantain peel powder reinforced polyethylene composite |
| A15 | 15 wt% plantain peel powder reinforced polyethylene composite |
| A25 | 25 wt% plantain peel powder reinforced polyethylene composite |

## 2. MATERIALS AND METHODS

### 2.1 Materials

Discarded bottle caps with resin identification code "2" were collected from refuse dumps and plastic waste collection centres in Zaria-Nigeria. They were washed; dried and shredded to particles of smaller sizes using Shredding machine WT600 model which constitutes the recycled (HDPE) matrix. Fresh unripe plantain peel (*Musa paradisiacal L*) was collected from local vendors in Zaria, Nigeria. It was washed and air-dried for 172 hours and finally sun-dried for 3 days. The plantain peel was finally pulverized into powdery form and sieved to 150µm particle size.

### 2.2 Methods

2.2.1 Modification of plantain peel powder (PPP)

Two grams of PPP was immersed in a 5% NaOH solution for 5 hours with continuous stirring. It was then rinsed and washed with water until the solution became neutral. The powder was filtered out and dried in an oven at 80 ℃ for 5 hours for further use in accordance with the procedure of Usman *et al*. (2016).

2.2.2 Preparation of PPP-Recycled high density polyethylene composites

The recycled high density polyethylene composites were prepared by melt mixing and compression moulding on a two roll mill and compression moulding machine respectively. The composite samples were produced by the addition of the shredded recycled HDPE while the rolls were in counter clockwise motion for a period of 10 minutes at a temperature of 160 ℃. Upon achieving a paste like matrix; the filler material (plantain peel powder) was introduced by gently applying manually as the rolls rotate at a rate of 500 rpm. The percentage fibre loading was varied from 0-25% (0, 5, 10, 15, 20 and 25%) respectively, in accordance with the work of Chike (2014).

2.2.3 Curing

The compounded samples were cured on a hydraulic machine 12000 model with electrically heated platens. The temperature of the platens was set at 150 ℃ and when the temperature was attained, the moulds were preheated to attain the platen temperature. The material was then cut to take the shape of the mould and was placed in between the platen with a pressure of 4 MPa for 10 minutes, and finally the cured samples were removed from the mould after cooling.

### 2.3 Physical and Mechanical Test

2.3.1 Density

The density of the composites was determined by measuring its respective mass and volume. Sample specimens of dimensions 20 x 20 x 5 mm were produced for the test in accordance with Dan asabe (2016). The mass was determined with the aid of a digital analytical balance ED 224S model to an accuracy of four decimal places. The volume of each sample was found using Archimedes's principle. The density was then calculated using equation (1):

$$\text{Density} = \text{mass/volume} \quad (1)$$

2.3.2 Water absorption

Water absorption test was carried out according to ASTM D570 (Klyovov, 2007) with oven dried specimen of

dimension 60 x 25 x 5 mm immersed in water at ambient temperature for 24 h. The specimen was removed and patted dry with a cloth (lint free) and then reweighed using a digital weighing balance ED 224S model. The dried weight before ($W_{initial}$) immersion and the weight after ($W_{final}$) immersion were noted. The percentage water absorption was determined using equation (2):

$$w_f - w_i / w_f \times 100 \quad (2)$$

2.3.3 Tensile test

The tensile testing of the samples was done at Engineering Materials Development Institute, Akure, Ondo State, Nigeria in accordance with ASTM D 638 (2014) standard. The samples were machined to dumb bell shape and then placed in Instron universal tensile testing machine 3369 model and the tensile strength and elastic modulus were evaluated.

2.3.4 Hardness test

The hardness test of composites is based on the relative resistance of its surface to indentation by an indenter of specified dimensions under a specified load. Samples of 30mm x 30mm x 4mm were tested for shore hardness values with a durometer Shore 'A' at the Materials Laboratory, Nigerian Institute of Leather and Science Technology (NILEST), Zaria-Nigeria. Five measurements were performed on the sample at different spots and the average of the values was taken as the hardness of the sample in accordance with *Usman et al.* (2016).

**2.4 Dynamic Mechanical Analysis (DMA)**

DMA was carried out using DMA 242E machine in strength of Materials Laboratory, Mechanical Engineering Department, Ahmadu Bello University, Zaria according to ASTM D7028 (2015). The test parameters: E', E" and tangent of delta (Tan $\partial$) were first configured via the Proteus Software using personal computer. Instrument set up included the sample holder (3-point bending), furnace temperature range of 30-110 $^0$ C, dynamic load of 4N, frequency range of 1-10 Hz and heating rate of 3K/min were configured. Sample dimension of 60 x 12 x 5 mm were produced for each test. The test specimens were loaded into the machine using a three- point bending and locked into the furnace.

**2.5 Scanning Electron Microscopy (SEM)**

Scanning Electron Microscope Pro X: Phenonm World 800-073 in the Department of Physics, Umaru Musa Yaradua University, Katsina-Nigeria was used to study the morphology of the composites. Samples imaging was done at accelerating voltage of 15kV at 500, 1000, 1500 and 2000 magnifications respectively.

**3. RESULTS AND DISCUSSION**

**3.1 Physical and Mechanical Properties**

3.1.1 Density

Figure 1 shows the density with increasing weight fraction of the fibre (reinforcement). A gradual increase in density could be observed up to 20% and sudden decrease at 25% weight fraction of fibre. The increase in density could be as a result of better interfacial adhesion between the fibre and the RHDPE matrix; while the decrease (above 20% weight fraction of reinforcement) could be attributed to increase in void content as the weight fraction of reinforcement increases.

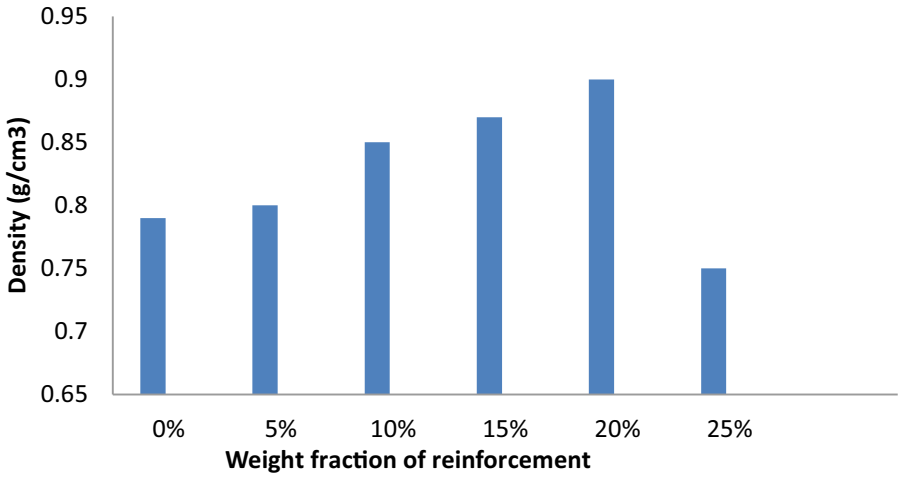



**Figure 1:** Effect of plantain peel powder on the density of RHDPE composites

3.1.2 Water absorption

Water absorption result presented in figure 2 shows that the percentage water absorption of the composites increases with weight of reinforcement and time, the highest being 3.4% at for 25% reinforcement at 192 hours. The increase may be attributed to increased surface area of the reinforcement in the RHDPE matrix, hence, leading to increased number of pores. The observed linearity of the plots beyond 192 hours (8 days), at all weight percentages of reinforcement indicates that there was no further increase in water absorption, which could be attributed to the fact that the pores created might have been saturated with water thus giving rise to such behaviour. Similar observation was reported (Agunsoye *et al*., 2012).

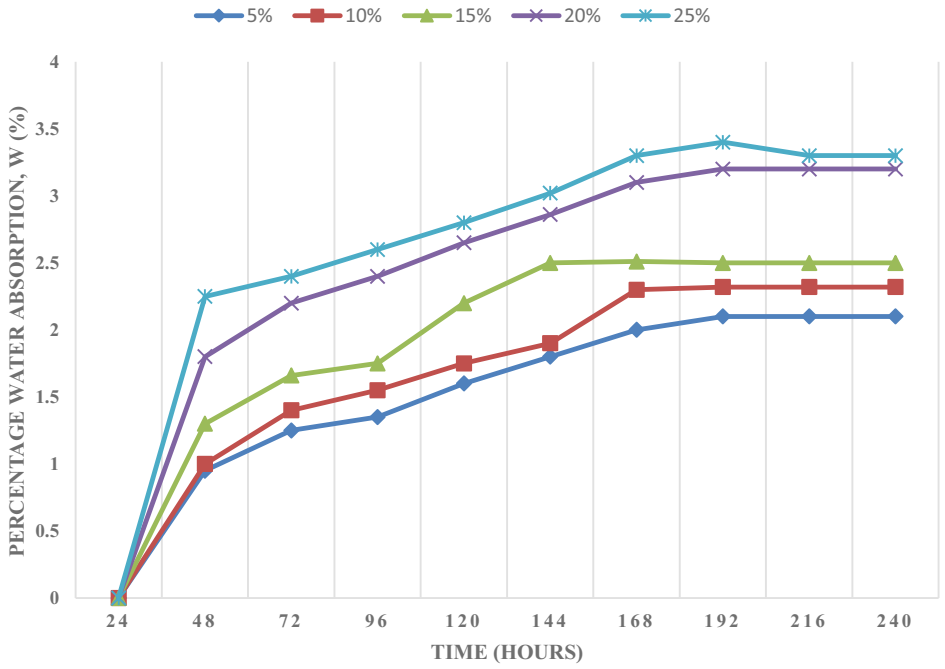



**Figure 2**: Percentage water absorption of PPP-recycled high density polyethylene composites

3.1.3 Tensile strength and elastic modulus

Figure 3 depicts the ultimate tensile strength (UTS) of the control and the composite with increasing weight fraction of the fibres. The increase in tensile strength of RHDPE composites with increase in PPP could be attributed to the stress transfer from the matrix. The ability of these fibres to support transferred stress likely came about as a result of better compatibility between the treated PPP and the polymer matrix (RHDPE).

The figure also shows the elastic modulus of the composites with weight fraction of fibre. The trend of the elastic modulus (stiffness) of the composites increases from 65.6 MPa to 90.7 MPa. An increase in elastic modulus with weight fraction of fibres has been reported by Dan asabe (2016).

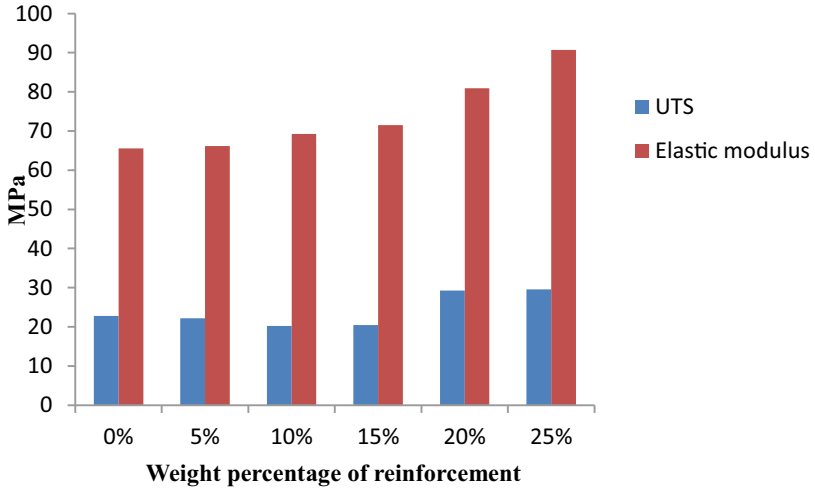



**Figure 3:** Effect of elastic modulus and UTS on weight percentage of reinforcement

3.1.4 Hardness (Shore A)

Graphical depiction of the effect of plantain peel powder on the hardness of RHDPE composites studied using Shore A durometer tester in accordance with ASTM D2240 is presented in figure 4. The hardness of the unreinforced RHDPE is 70.5 showing that the hardness of all plantain peel powder reinforced RHDPE increased with increase in the weight fraction of PPP incorporated into the polymer matrix. This implies that the presence of reinforcement may have improved the matrix surface resistance to indentation.

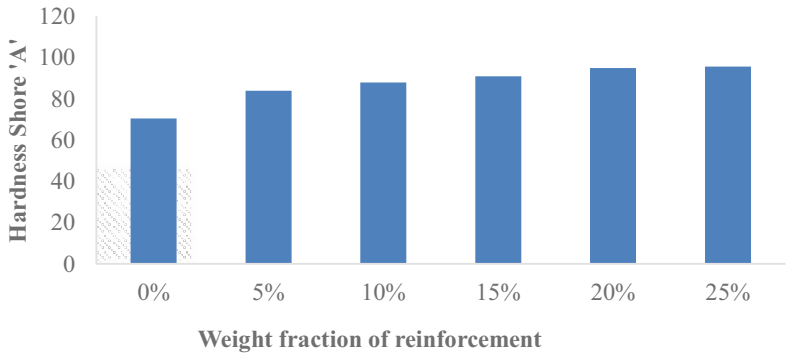



**Figure 5**: Hardness of PPP-recycled high density polyethylene composites

**3.2 Dynamic Mechanical Properties**

3.2.1 Storage modulus

Storage modulus (E') represents the elastic portion of materials and is defined as the maximum amount of energy stored by material during one cycle of oscillation (Gupta, 2017; Rana *et al*., 2017). It also gives an estimate of temperature-dependent stiffness behaviour and load-bearing capability of the polymer composites. Figure 6 below depicts the variation of E' with temperature for the control (unreinforced RHDPE) and treated PPP filled composites. It was observed that E' of all the composites increased with higher weight fraction of reinforcement across the entire temperature range due to enhanced stiffness, which is different from the control. This could be attributed to the fact that, when plantain peel powder was incorporated as a rigid reinforcement, the RHDPE component was embedded in the holes of the plantain peel powder, making them more closely connected and thus improving the deformation resistance of the composites.

Similar observation has been reported by other authors (Zhang *et al*., 2017; Jacob *et al*., 2018). With the higher weight fraction of PPP, the RHDPE dispersion in the composite was more homogeneous and the energy of the storage modulus increased. However, a decrease in E' was observed at elevated temperatures for all the composites due to loss in stiffness.

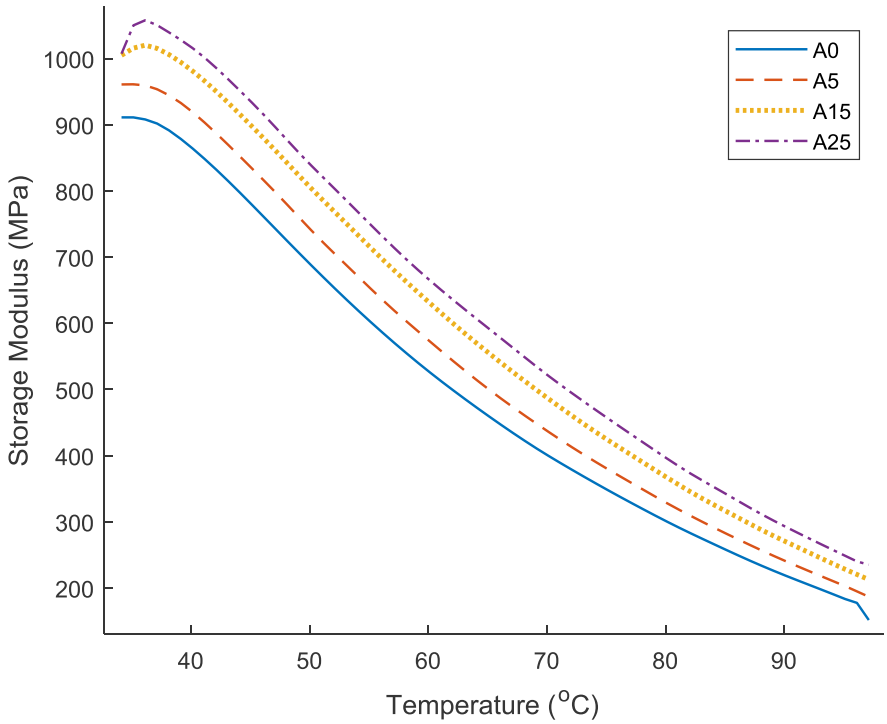



**Figure 6**: Variation of storage modulus with temperature of PPP composites at 1 Hz.

3.2.2 Loss modulus

Loss modulus (E") also refers to the imaginary part of complex modulus is defined as the amount of energy lost in form of heat by materials during one cycle of sinusoidal load (Gupta, 2017; Rana *et al*., 2017). It represents the viscous response of the polymer composites. The peak of loss modulus curve for polymer composites is known as dynamic glass transition temperature (Gupta and Bharti, 2017). The variation of loss modulus with temperature of PPP-reinforced RHDPE composites is shown in figure 7. From the

curve, it is evident that the loss modulus of the unreinforced RHDPE is higher than that of the composites which implies that incorporation of the filler increases the thermal stability of the composites. The observed decrease in loss modulus of the control sample from 53 to 63℃ indicates the transition of the material to its viscous state and that the energy dissipation of the material was rapid. The decrease in loss modulus with weight fraction of reinforcement has been reported by other authors (Palanivel *et al*., 2017; Gupta, 2018; Jacob *et al*., 2018).

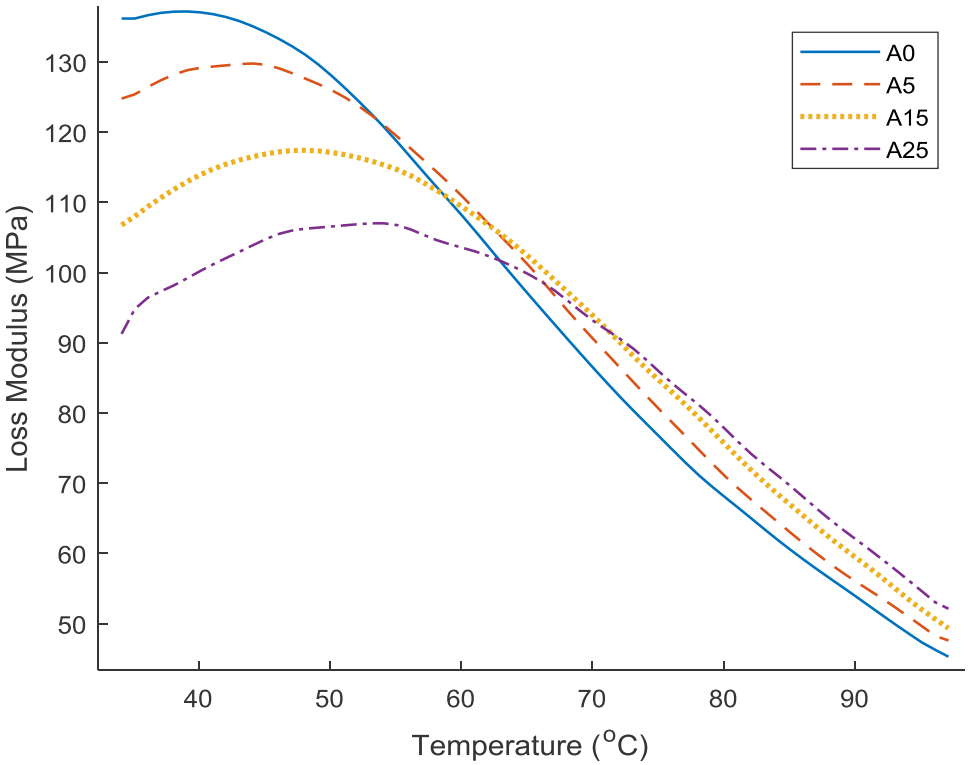



**Figure 7:** Variation of Loss modulus with temperature of PPP composites at 1 Hz.

3.2.3 Damping

Damping, (tan ∂) also called loss factor is the ratio of loss modulus to storage modulus and is related to impact resistance of materials (Gupta, 2018). Tan ∂ is expressed as a dimensionless number. A high tan ∂ value is indicative of a material that has a high, non elastic strain component, while a low value indicates one that is more elastic (Murayama, 1977). Graphical depiction of damping parameter with increasing weight fraction of reinforcement indicated decrease in loss factor of the composite (Fig. 8).

From the curve, it is obvious that the PPP-based composites showed a lower tan ∂ than the unreinforced RHDPE. This could be attributed to good interactions between the reinforcement (PPP) and the polymer matrix. Lower value of damping shows good load bearing capacity of the composites. The sharp rise in tan ∂ curve of the control sample at temperature above $95^0$C indicates that the energy dissipation of the material was rapid.

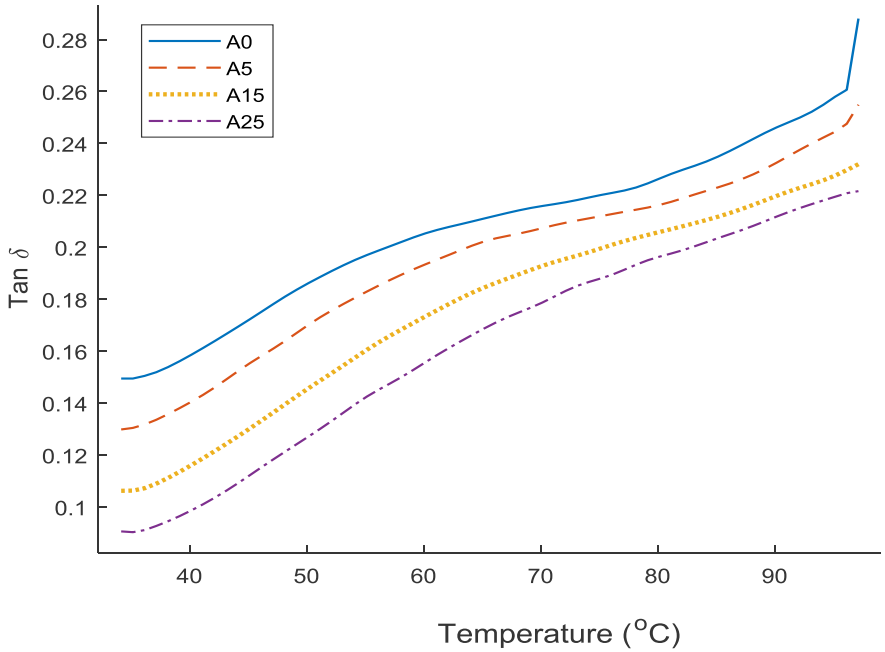



**Figure 8**: Variation of Tan ∂ with temperature of PPP composites at 1 Hz.

**3.3 Scanning Electron Microscopy (SEM)**

Plates (1-4) depict the microstructures of the plantain peel powder filled-RHDPE composites. Plate 1 reveals the presence of voids in the control (RHDPE) which was found to further increase with the incorporation of 5% PPP (plate 2). However, better distribution of PPP was observed with increase in weight percentage of reinforcement, leading to the voids considerably reduced. From the SEM results therefore, it can be deduced that the optimum thermo-mechanical properties observed at 25% weight fraction of reinforcement was as a result of better interfacial adhesion between treated PPP and RHDPE matrix. Similar observation was reported Jacob *et al*. (2018).

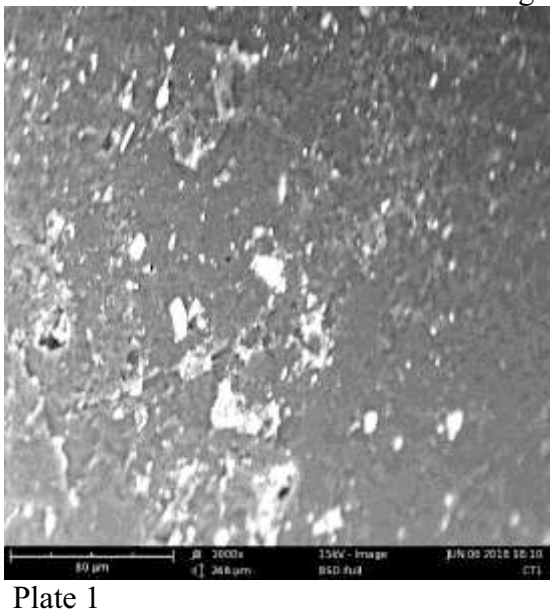


Plate 1

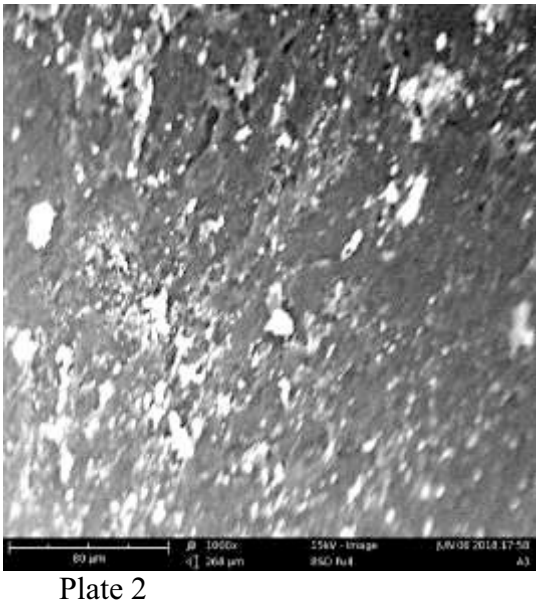


Plate 2

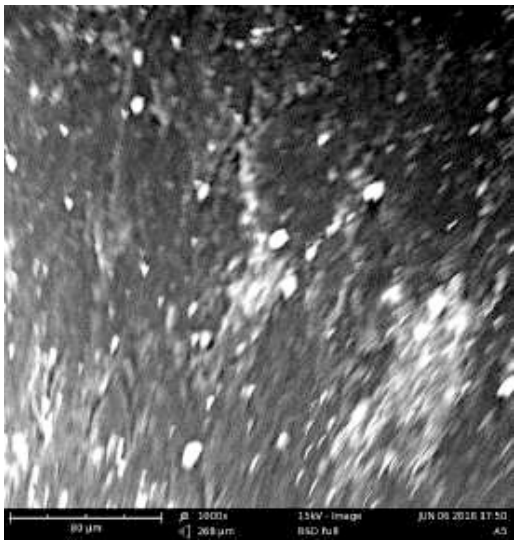
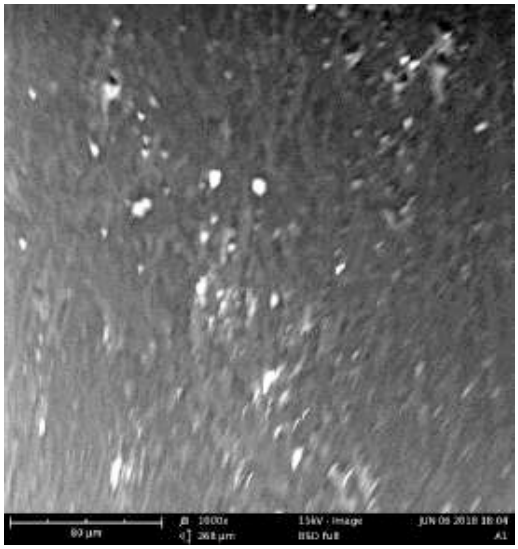


Plate 3



Plate

Plates 1- 4 show the SEM micrographs with (1) recycled HDPE at X1000; (2) 5% PPP at X1000 (3) 15% PPP at X1000; (4) 25% PPP at X1000, at accelerating voltage of 15kV.

## 4. CONCLUSIONS

From the results of this work, the following conclusions are made:

Incorporation of PPP into recycled HDPE improved its mechanical properties; optimum tensile strength of 29.7 MPa, stiffness of 90.7 MPa and hardness of 95.6 Shores at 25% weight of reinforcement. Similarly, Dynamic mechanical properties results indicated that the storage modulus and load bearing capacity were found to be maximum for composite with 25% reinforcement while a decrease in loss modulus was observed with increase in weight percentage of reinforcement and temperature.

SEM results further affirms that the observed optimum thermo-mechanical properties at 25% weight fraction of reinforcement was as a result of increased better interfacial adhesion between the plantain peel powder and RHDPE matrix.

## ACKNOWLEDGEMENT

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# Evaluation and Modification of Agricultural Waste in the Development and Property Enhancement of Polymeric Engineering Wares

**Momoh, F.P* and Abiodun, A. O**
Department of Polymer Engineering Technology, Auchi Polytechnic, Auchi, Nigeria

**Abstract**
*Coconut Shell is a major agricultural waste and environmental nuisance agent in most part of Edo state where it is predominantly produced and marketed. This study therefore seek to exploit the evaluation and modification of coconut shell waste through property development using carbonisation process at varying temperatures of 300, 400,500, 600 and 700$^0$C for three hours each. Optimised properties were deployed in design and development of automobile accessory specifically vibration dampeners. The carbonised and raw fillers were ground and passed through 100 µm sieve to achieve the rated particle size. X-Ray Diffractometer (Shimadzu 6000 Model) and Scanning Electron Microscopy (SEM) were used to evaluate and monitor changes in structural built up as indicated in the crystalline and mechanical behaviour of the carbonised fillers in the polymeric composites. The X-Ray Diffraction patterns and SEM images clearly showed that structural modifications were effected in the carbonised waste. Optimum performance were observed at carbonisation temperature of 500$^0$C. Results achieved are significant at developing the mechanical properties of polymeric matrix for use in engineering wares.*

**Keywords:** agro-waste, carbonisation, crystallinity, mechanical properties and morphology

## 1.0 Introduction

Environmental waste management and the increasing demand in the inclusion of local contents in Polymer product designs has become the impetus in recent technological advances and this approach has become a major cause for concern in polymeric development especially in the areas of engineering and technological applications. The extensive properties achievable in composite developments have made polymers and agricultural wastes attractive to researchers and product developers/designers. The inherent strength of polymers is of appreciable value, but yet modern trends in researches and product development consistently seeks a much more valuable use especially in the highly technical areas of applications. A major modifier needed in polymer products development is the filler class, especially from littered agricultural wastes in our immediate environment. Fillers of different colours, reinforcement and origin had played a major role in polymer modification over the years. A direct development and structural modification of fillers usually leads to indirect modifications in polymer networks, and hence a development in engineering wares that are polymer based (Momoh et al., 2016a).

Various work have been published on the application of agricultural based fillers in polymer composites such as pineapple, sisal, jute, cotton, bamboo, coconut shells, groundnut shells and wood flour fillers as reinforcement materials. Jacob et al, 2014 reported the evaluation of mechanical properties of coconut shell fibres as reinforcement material in epoxy matrix and established the possibility of using it as a new material for engineering applications. In 2011, Husseniyah and Mostapha reported the effect of filler content on properties of coconut shell filled polyester composites. Their results

* *Corresponding author: fridymoh@yahoo.com +2348034502422*

showed that the tensile strength, Young's modulus and water absorption of polyester/CS composites increased with the increasing CS content but elongation at break decreased.

Onyeagoro, 2012 studied cure characteristics and physical-mechanical properties of carbonised bamboo fibre filled natural rubber vulcanisates. Results obtained showed that compatibilized carbonised bamboo fibre filled vulcanisates exhibited improvement in the cure properties investigated over the non- compatibilized vulcanisation.

Bhaskar and Singh, 2013 carried out a study on physical and mechanical properties of coconut shell particle reinforced epoxy composite and reported that the mechanical properties- tensile strength and modulus of elasticity were closely related to physical property-density.

Shuaibu and Mamza, 2016 worked on the characterisation of polypropylene filled composite using scanning electron microscopy and x- ray diffraction; the result revealed that the crystallinity of the neat Polypropylene decreased with increasing filler loading and the compatibility of phases were adequate.

Also Sumari et al, 2013, studied the effect of ultrasound treatment on the morphology, particle size crystallinity, and crystallite size of cellulose and showed morphological breakdown with increase in ultrasound. Other researched works on modification of basic mechanical properties through carbonisation are that of Momoh et al, 2016a and Momoh et al., 2016b.

The present study report the removal of agricultural wastes that usually littered the bio-environment and the subsequent conversion of waste via modification through carbonisation. Possible application in engineering design and property development was evaluated. Scanning electron microscopy and x-ray diffraction were done. Mechanical and chemical properties of the composites were also evaluated.

## 2.0 Materials and Methods

### 2.1 Materials

Coconut shell wastes were found in Auchi, Edo State (Nigeria) and its environs. The natural rubber (Standard Africa Rubber, SAR3) was the grade used. Compounding additives: Zinc oxide, Stearic acid, Sulphur, MBTS, TMTD and Mineral oil were of commercial grades.

### 2.2 Preparation of the Waste and Carbonisation

The coconut shell waste was collected, washed in water to remove sand and debris, and then oven- dried at $95^0$C for 1 hour to remove moisture. 5 portions of 200g of the dried portions were carbonised at 300, 400, 500, 600 and $700^0$C for 3 hours each (Momoh et al., 2016a). The 5 carbonised portions and a raw portion were ground using a grinding mill to fine powder from which 100µm particle sizes were characterised using standard methods prior to compounding.

### 2.3 Compounding and Curing

A laboratory two-roll mill (180 x 360mm) was used for the homogenisation and mixing in accordance with ASTM – D3182. The nip gap, mill roll speed ratio of 1:1.25, sequence of addition and time of mixing of the additives were held uniform for the entire composite samples. The temperatures of rolls were maintained at $70^0$C. Maturation for 24 hours at $32^0$C was allowed for the selected compounds before press curing in accordance to ASTM – 1632-07. A compression pressure of 150kg/cm$^2$, temperature of $150^0$C and a time of 15 minutes were used as determined from ODR 2000 Model Rheometer.

### 2.4 Morphological Evaluation

Micro structural analysis was performed using a scanning electron microscope (FEI Inspect S-50 that using data analysis software server XT microscope). SEM magnification was at 1500X with a resolution of 80µm. The sample surfaces were coated with a thin layer of gold using a Bal-Tec SCD 005 sputter coater to provide electric conductivity. Secondary electron mode at a 10kv mapping acceleration voltage with full BSD was used. Particle