

## Development and Characterization of Hybrid Particulate-fiber Reinforced Epoxy Composites

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### Abstract

Although considered wastes, animal fibers and gastropod shell particles are biodegradable, have low density, high stiffness, considerably high impact absorption capacity and relatively low cost. Therefore, they are finding increasing use as reinforcement materials in polymer composites. This research work studied the tensile, hardness, and wear resistance properties of hybrid snail shell (SSP) and chicken feather barb fibers (CFB) reinforced epoxy composites. The stir cast molding technique was utilized to synthesize the composite samples with 3, 6, 9, 12, 15, and 18 wt.% of the hybrid SSPs/CFB. Compared with the control samples, SSP/CFB hybrid reinforcements enhanced the mechanical properties of the composites. Composites with intermediate weight fraction of 9 wt.% SSP/CFB exhibited overall optimum properties when benchmarked against the control sample with approximately 37, 37, 133, 19, and 59% improvement in wear, hardness, impact, and ultimate tensile strength properties respectively. These enhancements suggested a synergistic effect of the two reinforcement phases. The results presented in this study demonstrated the potential of utilizing bio-derived waste materials for synthesizing eco-friendly composites.

**Keywords:** Bio-derived reinforcement, Hybridization, Polymer composites, Recycling

### 1 Introduction

Polymer composites find applications in different engineering fields. The bulk of the reinforcement materials come from synthetic sources, which are mainly derived from limited sources of petroleum. With petroleum reserves declining and the upsurge in international awareness and efforts to minimize carbon emissions, there have been concerted research

efforts to utilize reinforcement materials derived from renewable and environmentally friendly sources, such as plants, animals, and their by-products [1]–[4]. Moreover, agro-derived matrix and reinforcement materials are projected to decrease the environmental footprint of composites.

Other than the eco-friendliness of natural fibers, they also possess some other advantages over synthetic/conventional fibers. These include biodegradability,

low cost, low carbon emission, less abrasive damage to processing plants, low density, and a means of economic empowerment for low-income farmers, especially the turning of agricultural wastes into useful technological materials [5]–[9]. Due to the increasing human population and demand for food, intensive agriculture is practiced in many parts of the world with the attendant huge agro-waste generation. Disposing of this huge amount of waste is challenging, as incineration contributes to global warming while landfill sites are overflowing. One approach to mitigate these challenges is to utilize these agro-wastes in a more sustainable manner as construction materials [5], [10].

Nowadays, consumers and many countries are at the forefront of demanding manufacturers to consider the environmental impact of their products. Therefore, different labels, such as eco, green, etc. are being attached to many products. Consequently, renewable fibers that are sourced from plant and animal wastes or by-products are now increasingly being investigated as candidate reinforcement phases in polymeric composites. These include plant-based fibers, such as cellulose [2], [3], *Cissus quadrangularis* stem fiber [4], bagasse [11], [12], grass [13], palm kernel shell [14], [15], coir fiber [16], [17] and animal-based fibers, such as wool [8], chicken quills and feathers [5], [7], [10]. The poultry industry generates several tons of waste, which includes feathers, blood, legs, bedding/litter materials, eggshells, and bones. Improper disposal of these by-products provides a good environment for disease vectors, insects, vermin, and other microorganisms that are harmful to man, livestock, and the environment [18], [19]. Therefore, it is imperative to properly manage these by-products. One promising approach is to utilize these waste materials, such as feathers, in the manufacture of value-added products, such as composite materials [5], [10].

Despite the alluring advantages of natural fibers, some challenges are encountered in utilizing them for synthesizing polymer composites. Chiefly among these challenges are high moisture absorption, low durability, variability in properties, and poor interfacial compatibility [1], [6], [7]. Therefore, these fibers are subjected to some treatment processes, such as alkali or mercerization [1], [12], [17], to eliminate some deleterious components of the fibers and enhance

the interfacial bonding strength. Additionally, the mechanical properties of the resulting composites are also improved [12], [20], [21].

Some research efforts have demonstrated the potency of chicken feather fibers (CFB) as suitable reinforcement materials. In a study on CFB-reinforced high-density polyethylene composites, authors report that alkaline treatment of the fibers improves the flexural and tensile properties of the composites [5]. Similarly, in the evaluation of the mechanical properties of CFB and quills reinforced vinyl ester and polyester composites, Uzun *et al.*, [10] document improved impact properties in the composites. Furthermore, Baba and Özmen [22] show that PLA composites exhibit enhanced mechanical properties due to the addition of CFB. Other than mechanical investigations, CFB has also been reported as an effective thermal insulation material in a polyester matrix [23] and a potential material for fabricating printed circuit boards in an epoxy matrix [24].

Similarly, researchers have investigated the viability of snail shell particulate (SSP) polymer composites. Snails and other mollusk shells are rich in calcium carbonate minerals [25], [26], which are important polymeric reinforcement and filler materials [17], [27], [28]. SSPs have been investigated in various polymer matrices, such as polyethylene [29], epoxy [30], [31], and polypropylene [25], [26]. In a study on recycled waste plastic composites, authors report enhancement in the mechanical and water absorption properties with the addition of SSPs [32]. Additionally, some studies on epoxy composites show that SSPs can be used to raise the glass transition temperature of the composites [30], while simultaneous enhancement in the thermal and mechanical properties of polypropylene composites is reported [25].

To further enhance the properties of interest in some polymeric composites, researchers have hybridized various forms of reinforcement. Therefore, this technique synergizes the advantages of each reinforcement material to maximize their performance [12], [33]. Consequently, hybrid CFB and carbon residues have been used to enhance the mechanical properties of epoxy composites [34]. Further, Gbadeyan and co-workers report upscaling of tensile, flexural, and water absorption properties in hybrid eggshell/SSPs-reinforced epoxy composites [31]. As research continues to grow in this field, various agro-wastes,

with the potential to be utilized as reinforcements, need to be investigated. Additionally, no prior study has investigated the hybridization of CFB and SSP as reinforcements for epoxy-based composites.

Therefore, in this study, we investigated the effects of hybrid snail shell particles (SSP) and chicken feather barb fibers (CFB) on the microstructural, tensile, hardness, impact, and wear resistance of epoxy composites. This work contributed to knowledge in waste management and sustainable production of reinforcement materials for fabricating polymeric composites. Additionally, we showed that these bio-derived hybrid reinforcement materials, at an optimal combination, had a positive impact on the assessed properties. The results presented in this study demonstrated the potential of utilizing bio-derived waste materials for synthesizing eco-friendly composites with potential applications in the exteriors (e.g., covers for side-view mirrors, bumpers and roof rails) and interiors (e.g., dashboard) of automobiles, crash helmets, etc.

## 2 Materials and Methods

The matrix material was an epoxy resin (Bisphenol A, A331) and amine hardener (A062), which were procured from Malachy Enterprise, Lagos, Nigeria. The reinforcement phases comprised snail shell waste (common African land snail, *Achatina fulica*) and chicken feather barb fibers. The snail shell wastes were sourced locally from a neighborhood market, while the chicken feather barb fibers were collected from the Teaching and Research Farm (Poultry Section) of the Federal University of Technology, Akure.

### 2.1 Preparation of snail shell particles (SSP)

Snail shell particles (SSP) were prepared following the protocol described in a previous study [35]. At first, the shells were cleaned to remove organic and other debris and put in an oven (KX350A; KENXIN International Co. Ltd., China) at 80 °C for 5 h to remove moisture from the shells. The shells were then pulverized to obtain large lumps of about 3–10 mm, which were later reduced to micron-sized particles by grinding in a ball mill. The resulting particles were sieved using a 65 µm sieve and further dried at 105 °C for 3 h to obtain SSP (Figure 1).



**Figure 1:** (a) Dried chicken feather barbs (CFB) (b) CFBs prepared for composite production (c) shell wastes from African land snail (*Achatina fulica*) and (d) micron-sized snail shell particles (SSP).

### 2.2 Preparation of chicken feather barb fibers (CFB)

The locally sourced chicken feathers were cleaned with warm water and detergent to rid them of dirt. They were oven-dried at 80 °C for 5 h. From these dried chicken feathers, quills and barbs were separated, as shown in Figure 1. Here, our research work focused only on the barbs of the feathers (CFB) to produce the hybrid composite material. The CFBs were further treated in 0.1 M NaOH to increase their surface roughness for proper anchorage in the matrix, increase interfacial bonding strength, and enhance the mechanical properties of the resulting composites [12], [20], [21]. Alkali treatment was carried out in a shaking waterbath operated at 50 °C for 4 h. Then, the treated CFBs were washed with copious amounts of distilled water to obtain a neutral pH. The resulting treated CFBs were dried in the oven at 80 °C for 24 h.

### 2.3 Fabrication of the hybrid SSP/CFB reinforced epoxy composites

SSP were dispersed in the epoxy matrix by stir-casting method, while the CFBs were hand-laid in a mold. Epoxy and hardener were mixed in the ratio of 2:1, while the mass ratio of SSP to CFB was fixed at 9:1. The composites were left to cure for 24 h in an ambient

atmosphere. To assess the optimal reinforcement weight fraction, wt.% (fraction of reinforcement mass to the total mass of the composite), the composites were produced by dispersing 3, 6, 9, 12, 15, and 18 wt.% of the hybrid SSPs/CFB, as shown in Table 1.

**Table 1:** Mass ratio of hybrid SSB/CFB reinforced epoxy composites

Composite Weight Fraction (%)	Epoxy Resin (g)	Hardener (g)	SSP (g)	CFB (g)
Control	200	100	-	-
3	194	97	8.1	0.9
6	188	94	16.2	1.8
9	182	91	24.3	2.7
12	176	88	32.4	3.6
15	170	85	40.5	4.5

## 2.4 Characterization and evaluation of the developed composites

### 2.4.1 Microstructural characterization

The surfaces of the samples were polished for microstructural examination. Scanning electron microscopy (SEM) Images were taken on a JEOL, JSM-IT300 (Tokyo, Japan) equipment in a low vacuum of about 100 Pa without the need for gold or platinum sputtering.

### 2.4.2 Wear test

The samples were assessed for wear resistance on a Taber abrasion tester (TABER Rotary platform abrasion tester - Model 5135, USA). Test samples had a diameter of 100 mm, a thickness of 6.35 mm, and a center hole of  $\varnothing 10$  mm, which was used to affix them during testing against two rotating abrasive wheels (thickness = 12.6 mm and diameter = 50 mm) at 500 rpm for about 1000 cycles. Wear resistance was measured as mass loss according to the ASTM D4060-10 standard [35] at a specified number of abrasion cycles. The wear indices were calculated based on the sample mass loss as shown in Equation (1).

$$\text{Wear index} = (m_i - m_f) \times \frac{1000}{k} \quad (1)$$

where,  $m_i$ ,  $m_f$  and  $k$  are the initial mass, final mass, and number of test cycles, respectively.

### 2.4.3 Tensile test

Samples for tensile testing were 115 mm long and 3 mm thick. Tests were carried out at room temperature on a universal testing machine (UTM, FS 300–1023, USA) at a crosshead speed of 5 mm/min based on the ASTM D-638-14 standard.

### 2.4.4 Impact test

Impact test specimens of dimensions  $64 \times 11 \times 3$  mm<sup>3</sup> were notched at the center and tested for notched Izod impact test following ASTM Standard D256 – 04 2004 [36]. The test was carried out using a Hounsfield balanced impact testing machine (model number h10-3) with a hammer that delivers 65 J of energy. Samples were placed horizontally on supports with a span of 60 mm.

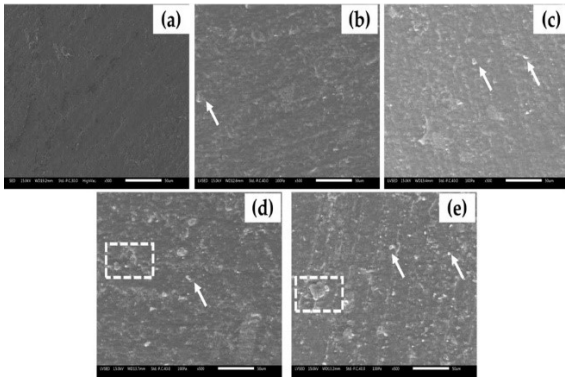
### 2.4.5 Hardness test

Samples were subjected to a hardness test based on the ASTM D2240 standard test method using a digital Shore D hardness tester. The indenter is the cone of 30° with a tip radius of 0.1 mm. A minimum of six indents were made and the average was reported as the hardness value for each sample.

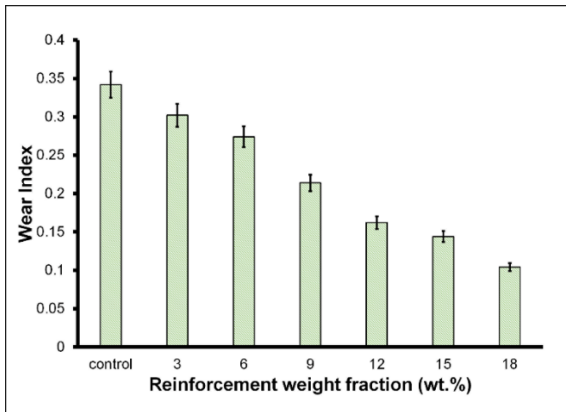
## 3 Results and Discussion

### 3.1 Microstructural characterization of the developed composites

Figure 2 shows the microstructural SEM images of the polished surfaces of the samples. In Figure 2 (a), the microstructure of the control sample is largely flat with minor striations. The reinforcement phase can be clearly seen in Figure 2(b)–(e). SSPs are identified by the white arrows and are largely uniformly distributed in the matrix. A homogeneous distribution of the reinforcement phases improves enhances the properties of the epoxy composites, as further expatiated in the sections below. As the weight fraction of SSPs increases in the matrix, some agglomerates could be seen in the Figure 2(d) and (e), which are the samples with SSP/CFB of 9 and 18 wt.%, respectively. These agglomerates are expected to have some negative effects on the mechanical properties of the composites,



**Figure 2:** Microstructural images of the epoxy composite samples (a) control (b) 3 wt.% SSP/CFB (c) 6 wt.% SSP/CFB (d) 9 wt.% SSP/CFB (e) 18 wt.% SSP/CFB. Scale bar is 50  $\mu\text{m}$ .



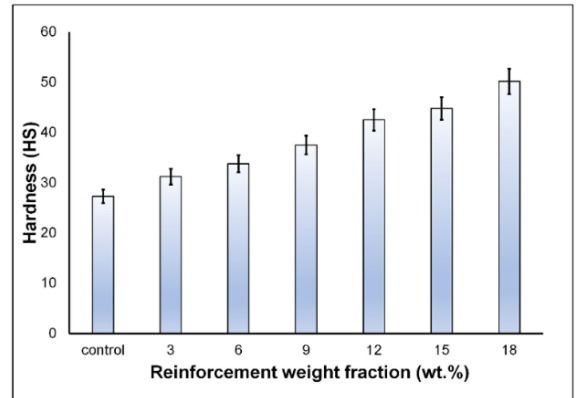
**Figure 3:** Effect of SSP/CFB hybrid reinforcements on wear index.

as observed in the mechanical characterizations and in some previous reports [30], [34], [37].

### 3.2 Wear index

Figure 3 shows the effects of hybrid SSP/CFB reinforcements on the wear index of the epoxy composites from the wear mass loss. It can be observed from Figure 3 that the greatest mass loss is associated with the control sample as compared to the other reinforced composites. Furthermore, resistance to wear increases as the reinforcement weight fraction increases.

Among the evaluated weight fractions, a sample with 18 wt.% SSP/CFB possesses the maximum wear resistance property with a wear index value of 0.104.



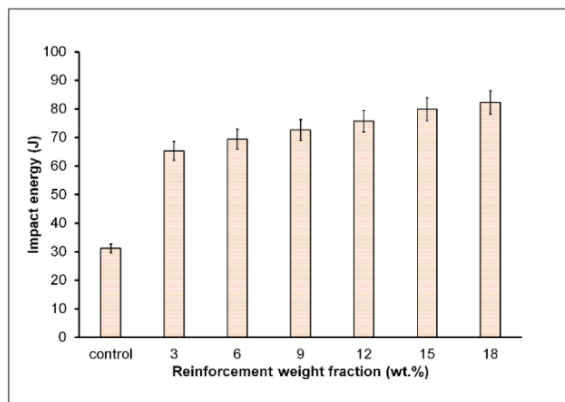
**Figure 4:** Effects of SSP/CFB hybrid reinforcements on hardness property.

This value is about 69.59% lower than the control sample. Thus, the SSP/CFB hybrid reinforcements constantly improve the wear resistance of the composites. SSPs are predominantly calcium carbonate, which is hard and strong, and have been successfully used to enhance the mechanical properties of various polymer matrices, such as polyethylene [29], epoxy [30], [31], and polypropylene [25], [26], which are applicable as polymer bearings, seals, and tools. Further, the increment in wear properties may also suggest there is a good interfacial interaction between the reinforcing phases and the matrix [32].

### 3.3 Hardness property

The resistance of an engineering material to permanent deformation is described as its hardness [4]. Figure 4 shows the influence of SSP/CFB hybrid reinforcements on the hardness of the composites. Compared with the control sample, the hardness values of the reinforced samples are higher, and this property shows consistent enhancement with increasing weight fraction of SSP/CFB reinforcements.

At only 3 wt. %, hardness improves by ~15%, with a further upscaling to 84% at 18 wt.% SSP/CFB. These enhancements in the resistance to the surface indentation in the reinforced composites can be rationalized based on the near homogeneous dispersion of both SSPs and CFB fiber in the matrix [38], high hardness of SSPs [30], and good interfacial adhesion between the fibers and the matrix [8,32]. A previous work on epoxy composite reinforced with Cissus



**Figure 5:** Effect of SSP/CFB hybrid reinforcements on impact energy.

quadrangularis stem fiber/red mud filler reported improved hardness properties [4].

### 3.4 Impact energy

Engineering materials are required to be tough to withstand service conditions. Therefore, impact energy, which is evaluated as the amount of energy required to fracture a sample, is an important engineering parameter. This, however, is influenced by the matrix characteristics, reinforcement morphology, and crystallinity of both the matrix and reinforcement phase [1]. A plot of the impact energy absorption of the composites is depicted in Figure 5.

Impact energy is lowest in the control sample but highest in the 18 wt.% reinforced composite. There is a significant improvement from the 31.2 J of energy absorbed by the control sample to 65.3 J in the 3 wt.% sample. This represents over 100% enhancement. Values of impact energy continue to rise with increasing reinforcement weight fractions although not as dramatic as between the 3 wt.% sample and the control sample. The presence of the SSP/CFB fiber hybrid reinforcement enhances the impact energy of the matrix. Epoxy has a weak impact strength, which can be enhanced by incorporating strong reinforcement phases in it [39]. SSP are stiff particles, which can minimize crack propagation, and thus enhance the impact energy of the composite samples [39]. As for the leveling-off in the impact energy, this may be attributed to some agglomerates observed in the SEM images (Figure 2(d) and (e)) of the samples with high

weight fractions of SSPs.

### 3.5 Tensile properties

A summary of the results of the tensile test of the samples is presented in Table 2.

**Table 2:** Tensile properties of the samples

Sample	Ultimate Tensile Strength (MPa)	Tensile Modulus (MPa)	Ultimate Strain (%)
Control	25.13 ± 1.21	450.88 ± 6.26	3.22 ± 0.12
3	14.40 ± 0.58	364.24 ± 4.37	1.70 ± 0.05
6	22.98 ± 0.78	435.99 ± 0.26	2.33 ± 0.10
9	29.78 ± 0.80	495.88 ± 11.97	5.96 ± 0.22
12	28.98 ± 1.54	523.40 ± 11.72	4.59 ± 0.23
15	23.79 ± 1.09	440.85 ± 1.67	3.00 ± 0.15
18	21.98 ± 0.64	356.10 ± 4.07	2.19 ± 0.10

At a low concentration of SSP/CFB (up to 6 wt.%) and high weight fractions (15 and 18 wt.%), ultimate tensile strength (UTS) decreases. However, for 9 wt.% and 12 wt.% SSP/CFB samples, UTS improves by approximately 19% and 15% when benchmarked against the control sample. The decrease in the UTS may be attributed to the debonding of the SSP and CFB from the matrix and the agglomeration of SSP particles at high weight concentrations [40]. Debonding may be related to poor adhesion between the matrix and the CFB/SSP [4]. Agglomerated rigid SSP particles limit effective load transfer from the reinforcement to the matrix and may also act as stress raisers, thereby accelerating crack initiation and propagation in the matrix [28], [30], [40]. Furthermore, the irregular shape of the fibers may hinder the adequate transfer of stress from the matrix, which may have a negative impact on the UTS [10]. Similar observations have been reported in CFB/crumb rubber-epoxy, *Cissus quadrangularis* stem fiber/red mud-epoxy [4], and [34] and periwinkle/almond-polypropylene composites [26].

Composites with enhanced strength are required in several load-bearing applications, such as automotive, aerospace, construction, oil and gas, and marine industries [41]. As for tensile strain and tensile modulus, there is a similar trend as observed for UTS. Both modulus and strain reduce at both lower (3 and 6 wt.% SSP/CFB) and higher (15 and 18 wt.% SSP/CFB) weight fractions of the reinforcements. Similar

reasons as expounded for the UTS may be applicable. Enhancements in tensile strain and modulus are seen at intermediate weight fractions of 9 and 12 wt.% SSP/CFB.

#### 4 Conclusions

In this work, we explored the potential of utilizing agricultural wastes as reinforcement phases in a polymer matrix. Waste snail shell particles (SSP) and chicken feather barb fibers (CFB) were incorporated as hybrid reinforcements in an epoxy matrix through a stir-casting technique. Microstructural, wear resistance, hardness, tensile, and impact energy properties of the epoxy composites were investigated. A homogeneous distribution of hybrid SSP/CFB was observed at a low weight fraction, whereas agglomerates formed at a higher weight fraction of the reinforcements. All the mechanical properties investigated showed enhancement due to the existence of the hybrid reinforcement phases in the matrix. Wear resistance and hardness properties were most improved at 18 wt.% SSP/CFB with a wear index value of 0.104 and 50 HS, respectively. In addition, the highest tensile modulus of 532 MPa was exhibited by the SSP/CFB sample with 12 wt.%. In summary, optimal property enhancements were displayed at an intermediate weight fraction of 9 wt.% SSP/CFB. These enhancements suggested a synergistic effect of the two reinforcement phases. The results presented in this study demonstrated the potential of utilizing bio-derived waste materials for synthesizing low-cost eco-friendly polymeric composites. Future studies may consider investigating the thermal properties and effect of alkali treatment protocols on the properties of the composites.

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#### Author Contributions

A.D.A.: conceptualization, investigation, reviewing and editing; A.D.A, I.O.O., M.O.I., E.E.E. and L.N.O: investigation, methodology, writing an original draft;

I.O.O. and N.I.A.: research design, data analysis; A.D.A and I.O.O.: conceptualization, data curation, writing—reviewing and editing, funding acquisition, project administration. All authors have read and agreed to the published version of the manuscript.

#### Conflicts of Interest

The authors declare no conflict of interest.

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