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Original Research Article

Mechanical Characterization of Camel's Foot (*Piliostigma reticulatum*) Fiber - Reinforced Recycled Low Density Polyethylene (rLDPE) Composite

[1]Kolo, L.A.M., *[2,3]Hammajam, A.A., [2]Abba-Aji, M.A., and [2]Shettima, I.I.

[1]Department of Works, Physical Planning and Development, University of Maiduguri, Borno State, Nigeria.
[2]Department of Mechanical Engineering, University of Maiduguri, Borno State, Nigeria.
[3]Department of Mechanical Engineering, Nigerian Army University, Biu, Borno State, Nigeria.
*hammajam92@gmail.com; lawanali99@gmail.com

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ABSTRACT

A study on the mechanical characterization of camel's foot fiber (CFF) reinforced recycled low density polyethylene (rLDPE) composite was conducted. The fiber and the matrix were locally source within University of Maiduguri community. The composites were produced by compounding process accompanied by compression molding techniques with varying percentage proportion from 0 to 25 wt % fibers loadings. The tensile, flexural and impact tests were conducted in accordance with ASTM D-638, D-790 and D-156 respectively. The tensile strength test result shows that the tensile strength of the composite increases with increase in fiber loadings to optimum value of 18.27 MPa at 15 wt % fiber loading. However, further fiber loadings resulted in decrease in tensile strength of the composites. The impact strength of the composites increases from 1,515 to 1,722 J / m^2 at 0 to 5 wt % fiber loading respectively. Subsequent fiber loadings recorded progressive slight decrease in impact strength. The flexural strength of the composite also increased with fiber loading to maximum value of 207.03 MPa at 10 wt % fibers loading but subsequently reduces with further fiber loadings. The scanning electron microscopy micrograph suggested good interfacial fiber-matrix bonding at certain fiber loadings. The composite produced can be use in the production of hand washing containers based on the mechanical properties exhibited.

1. INTRODUCTION

Natural fibers are one of the abundant resources that exist in nature. They are easily decomposable, bio degradable, renewable and cost efficient. It can be extorted from various plant and animal resources and are used for reinforcement material in polymers for the growth of natural fiber composite (Jeyapragash *et al.*, 2020). Natural fibers represent a category of renewable resources and a new category of reinforcements and additives is emerging for materials based on polymers. The creation of composite materials using natural fibers, often referred to as environmentally friendly composites, has gained significant attention recently and driven by a growing awareness of environmental concerns. Natural fibers stand out as effective materials that can substitute synthetic counterparts and their associated products, particularly in applications where reduced weight and energy conservation are crucial. The utilization of natural fiber-reinforced polymer composites and resins derived from natural sources presents a substantial opportunity for replacing existing materials reliant on synthetic polymers or glass fibers (Sanjay *et al*., 2016).

Due to the dominating advantages of natural fibers such as biodegradability, eco-friendliness, nominal cost, low density and high specific strength, they are being used opposite to synthetic fibers in many industrial applications. Despite these advantages, they face some limitations such as higher moisture uptake, quality variations, low thermal stability and poor compatibility with polymeric matrix. To overcome these challenges, chemical treatment of these fibers were found to be most suitable method to improve the adhesion with polymers, increase their strength and water absorption resistance, and improves the composite properties (Parul and Gupta, 2020).

Low-density polyethylene (LDPE) is a thermoplastic that originates from the ethylene monomer. First produced in 1933 by Imperial Chemical Industries (ICI) using a high-pressure process through free radical polymerization, LDPE is not reactive at room temperatures except in the presence of strong oxidizing agents. Certain solvent can induce swelling. LDPE may endure heating up to the temperature of 176 ^{o}F consistently and 194 ^{o}F for a short duration. Available in opaque or translucent variations, LDPE is known for its remarkable flexibility and toughness and exhibits increased branching, carbon atoms of about 2 % (Dennis, 2010). LDPE materials are commonly use in various uses such as plastic packaging film, disposable plastic products, and pipes among other products. It is well - known that waste low-density polyethylene, particularly from pure water sachets presently plays a vital role in the provision of potable drinking water in Nigeria. However, these polyethylene sachets are non-biodegradable and constitute health and environmental hazard because they encourage the spread of water borne diseases, constitute dirty environment and can cause suffocation to small children and animals (Ahmed *et al*., 2022). In many developing countries low-density polyethylene sheets, bags, and water sachets are a major waste problem because local collection and recycling systems do not exist and as a result, LDPE has no value and is dumped causing aesthetic, environmental and public health issues (Jnr *et al*., 2018). This waste contributes significantly to solid waste accumulation and posing environmental challenges. Recycling of LDPE materials would addresses the challenges posed by these materials and also provides an eco-friendly alternative that supports waste reduction and promotes environmental sustainability.

Camel's foot tree (*Piliostigma reticulatum*) is a leguminous plant that falls under the family of Caesalpiniaceae, which includes trees, shrubs, or occasionally scramblers. Between November and April, this perennial tree produces white to pinkish petals. The fruits of the tree are hairy, hard, flattish pods that undergo a transition from a rusty brown color to a woody and twisted state. These pods split at ripening and are usually persistent on the tree, with fruiting occurring between June and September. In the Yoruba language in Nigeria, the seed is locally known as Abefe, while other names for the tree include Kalgo (Hausa), Monkey bread, Camel's Foot, Okpoatu (Igbo), and Kalurr in Kanuri. The Camel's Foot Tree flourishes in open woodlands and damp, wooded grassland areas found at low to moderate altitudes. Its distribution spans extensively across Africa and Asia. The plant grows abundantly in the wild, without cultivation, in various parts of Nigeria, including Zaria, Bauchi, Ilorin, Plateau, Lagos, and Abeokuta (Jimoh and Oladiji, 2005) and also in the study area, Maiduguri. By tapping into the potential of these fibers, it is possible to create composites that are not only effective in engineering applications but also align with principles of environmental responsibility and sustainable practices.

Numerous studies have delved into the characteristics of natural fibers employed in reinforcing polymer composite materials. Regrettably, limited data is available regarding the use of *Piliostigma reticulatum* fiber reinforced polymer composite. The literature reviewed thus far does not report any research on the utilization of *Piliostigma reticulatum* fiber to reinforced recycled low-density polyethylene (rLDPE) composites. Therefore, this research aims to address this gap by exploring the mechanical properties of rLDPE reinforced with *Piliostigma reticulatum* fiber.

2. MATERIALS AND METHODS

2.1. Materials

The materials used for this research work include the following: waste low density polyethylene (used pure water sachets) obtained within University of Maiduguri community; Bast fibre of *Piliostigma reticulatum* fiber sourced locally from around University of Maiduguri environment of Borno State; Sodium Hydroxide obtained from Hafason Chemicals and Scientific Equipment, Samaru Zaria, Kaduna State; Distilled Water obtained from Chemistry Laboratory, Nigerian Institute of Leather and Science Technology, Zaria, Kaduna State and Benzyl peroxide obtained from Chemistry Laboratory, NILEST, Zaria, Kaduna State; NaOH obtained from Chemistry Laboratory, NILEST, Zaria, Kaduna State of Nigeria.

2.1.1. Equipment used

The equipment used in this study are:

i. Two roll mill machines used for compounding the matrix and fiber to produce the composite sheet.
ii. Compression molding machine used for shaping and curing of the composite
iii. Resil impact tester machine used for determining the impact strength of the composite
iv. Universal material testing machine used for tensile strength test of the composite
v. Universal Testing Machine used for flexural strength test of the composite
vi. Digital Weighing Balance machine used for measuring the weight of the fiber and matrix used
vii. Scanning electron microscope machine used for SEM test

2.2. Methods

2.2.1. Collection and preparation of matrix

The matrix used for this research work was waste low density polyethylene (pure water sachets) shown in plate I. The matrix was source within University of Maiduguri community dumpsites. They were sorted, washed with detergent thoroughly and rinsed with water to get rid of dirt and unwanted materials on the polymer and then dried in enclosed environment.

Plate I: Waste low density polyethylene (pure water sachets)

2.2.2. Collection and preparation of camel's foot fiber

Fresh samples of *Piliostigma raticulatum* from its plant shown in plate II were sourced locally within University of Maiduguri community of Borno State, Nigeria and the bark were decorticated from the stem as shown in plate III to obtained fiber bunches shown in plate IV. These fiber bunches are subjected to water retting process for 21 days to produce the fiber strings shown in plate V. These fibers were subsequently thoroughly rinsed with distilled water to remove any undesired biological substances present on their surfaces. They were then distributed across a waterproof sheet and kept in a sheltered shed for drying.

Plate II: *Piliostigma raticulatum* tree

Plate III: Camel's foot tree stem

Plate IV**:** Camel's foot tree bast fiber bunch

Plate V: Camel's foot tree bast fiber string

2.2.3. Formulation of the composite material

The fiber and matrix formulations were varied by percentage proportion of 5 wt % fiber loading from 5 wt % to 25 wt % of fiber loading as reported by Jocob *et al.*, (2018) i.e. 5 wt %, 10 wt %, 15 wt %, 20 wt % and 25 wt % CFF loading and also a control sample of 100 wt % rLDPE composite. The fiber weight fraction (FWF) was computed from the fiber volume fraction using laminate formulae, Equation 1 (Mala *et al.*, 2018).

$$FWF = \frac{\rho_f \times FVF}{\left[\rho_m + \left(\left(\rho_f - \rho_m\right) \times FVF\right)\right]} \tag{1}$$

Where:

FVF = fiber volume fraction, %

ρ_f = density of fiber, g / cm^3

ρ_m = density of matrix, g / cm^3

The Mass of the matrix (control sample composite), Mm is calculated from Equation 2.

$$Mm = \rho_m \times V_{mold} \tag{2}$$

Where; Mm = mass of matrix (control sample composite)

ρ_m = density of matrix, g / cm^3

V_{mold} = volume of mould, cm^3

Also, the volume of the mould is computed from Equation 3

$$V_{mold} = \left(l \times w \times b\right) cm^3 \tag{3}$$

Where; l = length of the mould, cm
w = width of the mould, cm
b = breadth of the mould, cm
The mould used has a dimension of: l = 170mm; b = 120mm and w = 3.2mm
The mass of the fiber, M_f is obtained by using Equation 4;

$$M_f = Mm \times FWF \quad (4)$$

The mass of fiber and matrix for the varied proportions obtained using the above equations are presented in Table 1. Note that Density of matrix is 0.91 g / cm^3 and density of fiber is 1.00 g / cm^3 (Dimas *et al.*, 2020).

Table 1: Sample formulation

S/No	Sample	Fiber loading (%)	Mass of matrix (g)	Mass of fiber (g)
1	Control	0	59	0
2	A	5	55.77	3.23
3	B	10	52.58	6.42
4	C	15	49.42	9.58
5	D	20	46.28	12.72
6	E	25	43.18	15.82

2.2.4. Production of the composite material

A hot compression process was use to produced the composite samples in the Nigerian Institute of Leather and Science Technology (NILEST) recycling (polymer) workshop, polymer and leather waste recycling facility, NILEST Zaria, Kaduna state. The process involves the introduction of the polymer material while the rolls of the two rolls mill machine were in counter clockwise motion and soften for 5 minutes at a temperature of 170 °C according to the formulations presented in Table 1. Upon attaining a band and bank formation of the polymer on the front roll, the prepared *Piliostigma reticulatum* fiber was introduce gradually to the bank; cross mixed and permitted to blend for 3 minutes. The blend was flattened into sheets and accordingly labeled. These composite sheets obtained from the compounding process were placed into a metal mould of dimensions 170 mm x 120 mm x 3.2 mm and was placed on the Compression Molding Machine for shaping and curing at temperature of 150 ºC and pressure of 2.5 MPa for 5 mins. The composite was remove after cooling from the mould and labeled accordingly as shown in plate VI.

Plate VI: Designation of composite samples

2.3. Mechanical Properties Characterization

2.3.1. Tensile strength test

The tensile strength test were conducted following ASTM D-638 using Universal Material Testing Machine at material testing laboratory, Department of Metallurgical and Material Engineering, Ahmadu Bello University Zaria, Kaduna State. A dumbbell shaped specimen shown in plate VII obtained from the composite samples were subjecting to a tensile force and tensile strength, tensile modulus percentage elongation at break and other parameter. The results for the computer generate each specimen automatically, computed and the average results were computed accordingly.

Plate VII: Dumbbell specimen for tensile strength test ASTM D-638

2.3.2. Impact Strength test

The charpy impact strength test was carried out according to ASTM D-256 in the Instrumentation laboratory, NILEST polymer and leather waste recycling facility, Zaria using Resil impact testing machine. The specimen measuring 63.5 mm x 12.7 mm x 3.2 mm and 45^o notched at the middle of the test specimens for all the produced impact strength composite specimens as shown in plate VIII produce from the panel. The specimen was clamp vertically on the jaw of the machine and hammer of weight 1500 N was release from an inclined angle 150°. The impact energy for corresponding tested specimens is taken and recorded. Impact strength was computed accordingly using Equation 5.

$$\text{Impact Strength} = \frac{Average\ Impact\ Energy}{crossectional\ area\ of\ the\ specimen} \quad (J / m^2) \tag{5}$$

Cross sectional Area of the specimen = 8.0645 x 10^{-4} m^2

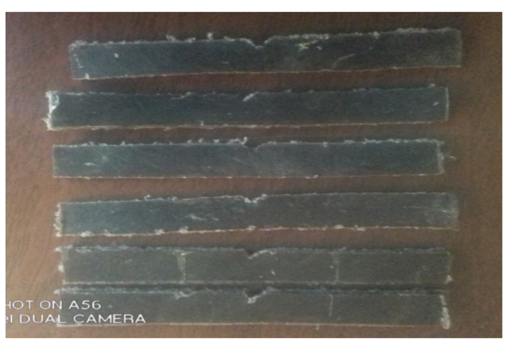

Plate VIII: Impact strength test specimen ASTM D-256

2.3.3. Flexural strength test

The flexural strength test on the blends was carryout according to ASTM D-790 in the Department of mechanical engineering, Ahmadu Bello University Zaria, Kaduna State using universal testing machine. The specimen measuring 76 mm x 25 mm x 3.2 mm shown in plate IX was placed on a support span horizontally at 80 mm gauge length and a constant load was exerted at the center by the loading nose, applying three-point bending, the sample specimen was subjected to induce failure. The optimal load (N) and its corresponding deflection (D) were then recorded as the sample specimen failed. The flexural strength was computed using Equations 6.

$$\text{Flexural Strength} = 3FL / 2bd^2 \text{ (MPa)} \quad (6)$$

Where,
F = Maximum Load at break
L = distance between the support spans at both edge of the specimen = 80 mm
b = Sample width = 25 mm
d = Sample thickness = 3.2 mm

Plate IX: Flexural strength test specimen ASTM D-790

3. RESULTS AND DISCUSSION

3.1. Tensile Strength Test Result

Figure 1 presents the result of the average tensile strength of the CFF / rLDPE composite. The results indicated progressive increased in tensile strength from 9.94, 10.05, 10.09 and 18.46 MPa for 0, 5, 10, and 15 wt % fiber loadings respectively. The optimum tensile strength was achieved at 15 wt % fiber loading. The addition of fibers enhances the tensile strength of the composite by providing additional reinforcement and resisting the applied tensile forces thereby distributing the load and improves the composite's overall strength.

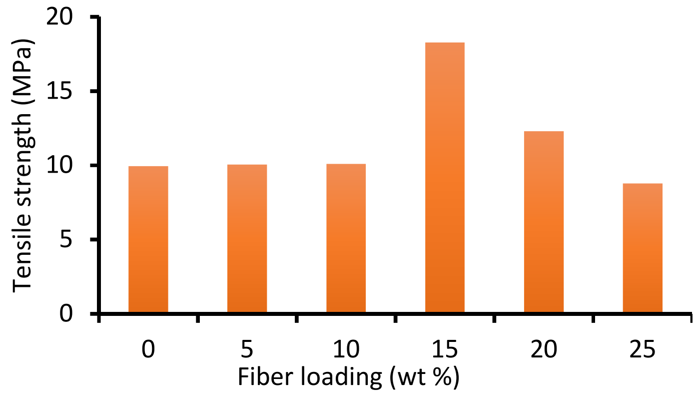

Figure 1: Effect of fiber loading on the average tensile strength of CFF / rLDPE composite

The SEM micrograph of 15 wt % fiber loading shown in plate X revealed better bonding between the matrix and the fiber. Further fiber loading reduces the tensile strength to 12.29 and 8.79 MPa at 20 and 25 w % fibers loading respectively. This could be attributing to fiber agglomeration at higher fiber loading, leading to stress concentrations and premature failure (Himanshu *et al.,* 2019). This result is in agreement with the work of Daramola, *et al.,* (2017). Also, the SEM micrograph at 25 wt % fiber loading of tensile test specimen shown in plate XI revealed fiber agglomeration, voids and fiber pull out due to poor matrix – fiber adhesion.

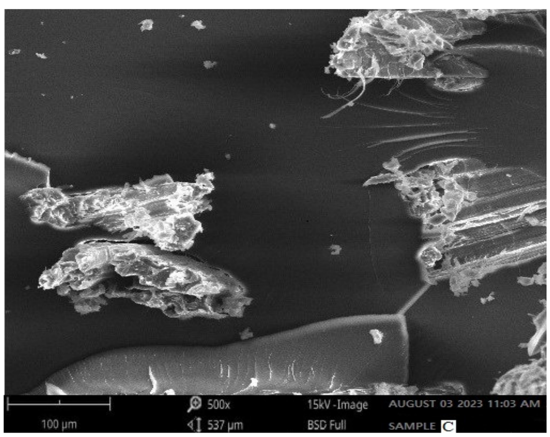

Plate X: SEM micrograph of 15 wt % fiber loading of tensile strength test specimen

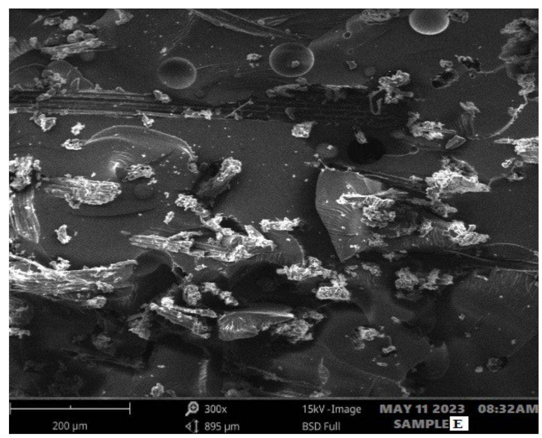

Plate XI: SEM micrograph of 25 wt% fiber loading of tensile strength test specimen

3.2. Flexural Strength Test Result

Figure 2 shows the result of average flexural strength of CFF / rLDPE composite. The result revealed increased in tensile strength with fiber contents up to 10 wt % from 132.81, 187.50 and 207.03 MPa (optimum flexural strength) at 0, 5, and 10 wt % fiber loadings respectively. The increased in flexural strength is primarily attributed to the reinforcing impact of the fiber, which enhanced the load-bearing capacity and stiffness of the composite and provide additional structural integrity to the composite, distributing the applied load more efficiently and reducing the susceptibility to bending-induced failure. The SEM result at optimum flexural strength (10 wt % fiber loading) shown in plate XII revealed homogeneity in matrix – fiber bonding. Further addition of fiber beyond this optimum value reduces the flexural strength to 99.99, 82.81, and 62.50 MPa at 15, 20 and 25 w % fiber loadings. The declined in flexural strength could be attributing to fiber entanglement, high fiber loading and increased brittleness, which negatively affects the flexural strength of the composite. Abdulrahman *et al*., (2015) and Daramola, *et al*., (2017) reported similar trends.

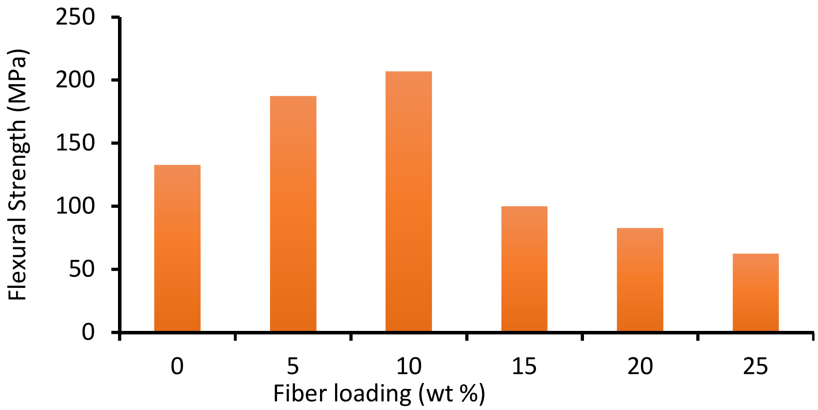

Figure 2: Effect of fiber loading on the average flexural strength of CFF / rLDPE composite

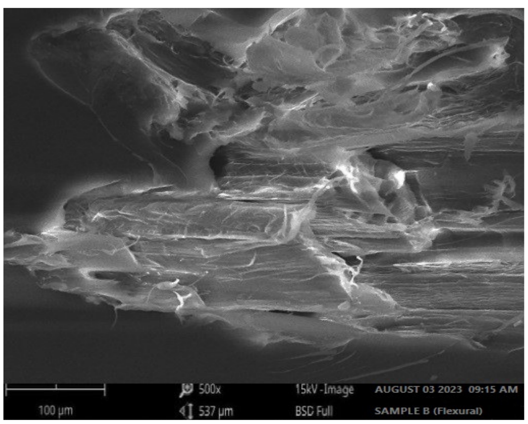

Plate XII: SEM micrograph of 10 wt% loading of flexural strength test specimen

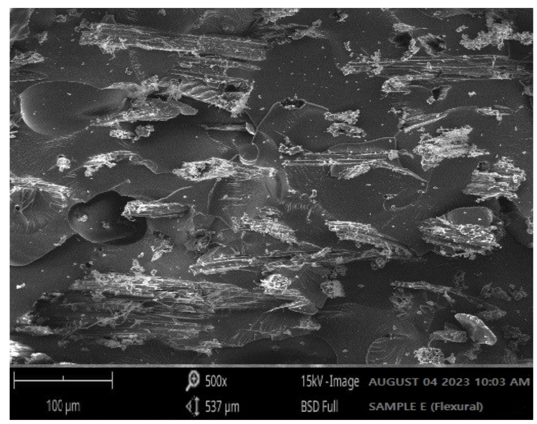

Plate XIII: SEM micrograph of 25 wt% fiber loading of flexural strength test specimen

The SEM micrograph of the 25 wt % fiber loading of flexural test specimen shown in plate XIII revealed voids due to air entrapment during curing and cooling of the composite, fiber agglomeration and fiber pull out which could be due to poor matrix – fiber adhesion.

3.3. Impact Strength Test Result

Figure 3 presents the result of average impact strength of the camel's CFF / rLDPE composite produced at different fiber loadings from 0 to 25 wt %. The result indicated increased in impact strength from 1,515 to 1,722 J / m^2 at 0 to 5 wt % fiber loading respectively. The increased of impact strength at this point could be attributed to the plasticity, less brittleness and flexibility nature of the composite at 5 wt % fiber loading which enables the absorption and efficient distribution of the impact energy, (Olusunmade *et al.,* 2016). In addition, SEM micrograph of the impact strength test specimen at 5 wt % fiber loading shown in plate XIV indicates good matrix – fiber adhesion which enhanced the absorption of the impact energy.

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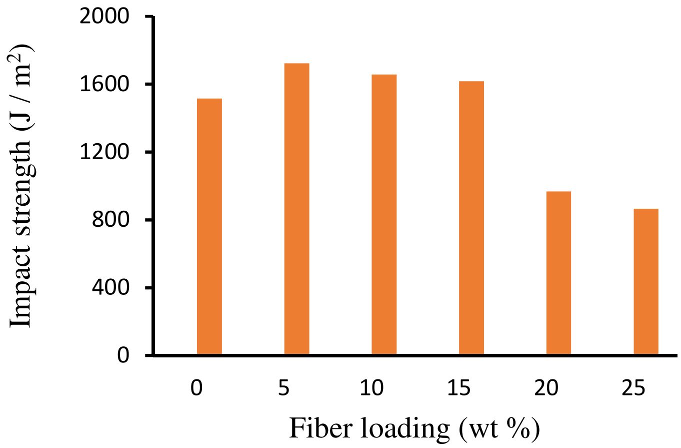

Figure 3: Effect of fiber loadings on the average impact strength of CFF / rLDPE composite

Further increase in fiber loading reduces the impact strength to 1,657, 1,618, 967 and 866 J / m^2 at 10, 15, 20 and 25 wt % fiber loadings respectively. The decreased in impact strength could be attributed to increased stiffness of the composite at increased fiber loadings. Also, the SEM micrograph of impact strength at 25 wt % fiber loading shown in plate XV revealed fiber entanglement and voids which indicates poor matrix – fiber adhesion. This result is also in agreement with the work of Hammajam *et al.*, (2017).

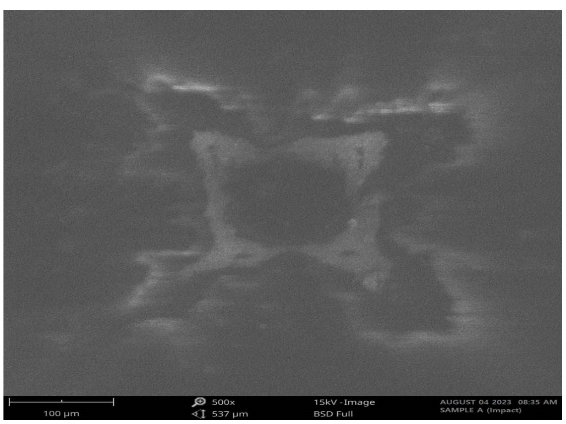

Plate XIV: SEM micrograph of 5 wt % fiber loading of impact strength test specimen

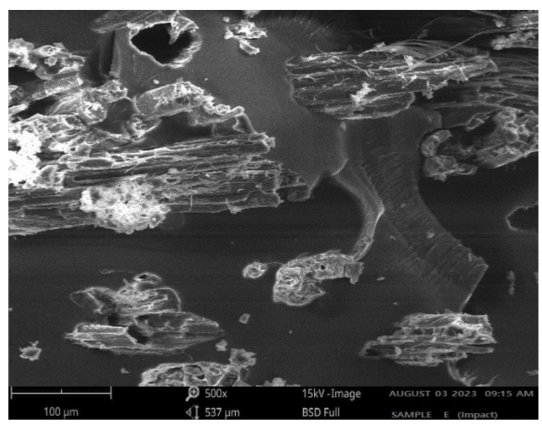

Plate XV: SEM micrograph of 25 wt% fiber loading of impact strength test specimen

4. CONCLUSION

The tensile strength test result indicated that the tensile strength of the material appreciated with addition in fiber loading to optimum value of 18.27 MPa at 15 wt% fibers loading. However, further addition of fiber results in composites' tensile strength reduction and a least value of 8.79 MPa at 25 w % fibers loading. The flexural strength of the composite also increased with fiber loading to maximum value of 207.03 MPa at 10 wt % fibers loading. However, subsequently reduces with further fiber loadings to a least value of 62.50 MPa at 25 wt % fiber loadings. The composites' impact strength increases from 1,515 to 1,722 J / m^2 at 0 and 5 wt % fiber loadings. However, subsequent fiber loadings shows progressive decrease with fibers loadings, yet above the control sample value up to 15 wt % fiber loadings. Minimum impact strength of 866 J / m^2 was recorded at 25 wt % fiber loadings. The SEM results shows that increased in fiber loadings beyond certain points leads to poor adhesion of the fiber and matrix which affects the mechanical properties of the material. The combination of *Piliostigma reticulatum* and waste pure water sachets is promising for composite development based on the good mechanical properties exhibited by the composite especially for producing hand washing containers.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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