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ORIGINAL RESEARCH ARTICLE

DEVELOPMENT AND CHARACTERIZATION OF GROUNDNUT SHELL POWDER-FILLED POLYLACTIC ACID (PLA) COMPOSITES FOR SUSTAINABLE ENGINEERING APPLICATIONS

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ABSTRACT

This paper explores the economic potential of using groundnut shell powder as a filler in biodegradable polylactic acid (PLA) composites. Groundnut shells, an abundant agricultural waste, are proposed as a low-cost reinforcement material to develop sustainable bioplastics. The investigation characterized and determined the mechanical properties of groundnut shell powder (GSP) filled PLA thermoplastic composites. Two different fiber sizes; 150 µm and 212µm were pulverized. The fiber loadings were 10 %, 20 %, and 30 % by weight. The GSP-PLA composites were prepared by use of internal mixer, followed by compression molding process. Tensile and Flexural properties were tested using universal testing machine (UTM). The tensile strength decreased from control sample (0 %) to 10 % by weight fiber loadings for all mesh sizes. In addition, the strength increased from 14.2 MPa and 15 MPa for 150 µm and 212 µm respectively, at a loading of 20%. The modulus increased from 712.9GPa for a control sample (0%) loading to 2344.9GPa at 30% loading for 150 µm. Furthermore, it increased to 1576 GPa for 212 µm. The tensile percentage elongation reduced at 10% loading compared to 0% control sample. Flexural strength and modulus increased for both mesh sizes at all the fiber loadings. However, at 30% fiber loadings, the flexural strength and modulus decreased slightly for 212µm size composites. The hardness of the composites increased for both mesh sizes at all the fiber loadings. It is presumed that the tensile properties performed good at 20% loading as deduced from the findings. while flexural properties increased for all loadings. The study identifies key application areas including packaging, construction, agriculture, and consumer products, highlighting their relevance in supporting eco-friendly startups and rural industries. The innovation aligns with global sustainable development goals (SDGs) number 7 and 8 and offers promising opportunities for green entrepreneurship, especially in agrarian economies.

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1.0 Introduction

The growing emphasis on environmental sustainability and circular economy principles has catalyzed the development of bio-based and biodegradable polymer composites. Biocomposite, polylactic-rich biomass material has emerged as promising reinforcement and offers versatile applications and potential benefits in various fields, especially in natural fiber composites (Ahmed *et al.*, 2022; Kolo *et al.*, 2022). The use of natural fibers for composite material manufacturing have received great attention because of their sustainability and renewability. Research and advancement on natural fiber composites are continually rising and their function is growing into novel fields of usage. The modern age of technology presents an abundant of bio-based composites. This gives better chance for environmentally friendly, lower energy utilization, insubstantial weight, lagging and sound absorption behaviors, reduction in volatile organic radiation, and decreased reliance on synthetic. Advancement of workable resources as the substitute for synthetic materials are being investigated to reduce the over-reliance on oil or petroleum (Mohammed *et al.*, 2022).

On the other hand, Agro waste cereals such as rice, barley, maize, sorghum, wheat etc contribute significantly as reinforcements or fillers in plastic composites productions in recent time (Hammajam *et al., 2019*). The importance of rice husk in composite was further discussed by Simone *et al*. (2022) which indicate the description of lignocellullosic rice husk filled with polyethylene composites. For example, steam explosion was employed for obtaining sweet sorghum fibers to be use as reinforcing agent in poly lactic acid base composites. Cascade mixer was employed for barley husk polypropylene composite by Andrzej *et al*. (2010) whereas Ricardo *et al*. (2023) uses thermogravimetric analysis of composite obtained from sintering of rice husk scrap tire mixture; and Ismail *et al*. (2003) employed effect of mixing sequence in the preparation of white rice husk ash filled polypropylene/ethylene propylene monomer composites. PLA is thermoplastic polymer with high strength and modulus that can be derived from annually renewable resources. This makes PLA very vital environmentally friendly biocomposites matrix. It can be degraded by simple hydrolysis of the ester bond and does not demand the presence of enzymes to catalyse (Ortega-Leyva, 2022). It also has melted and glass transition temperatures of 175 ^{o}C and 55 ^{o}C respectively. These attributes make it suitable candidate for most degradable natural fibers as environment friendly materials. Ahmed *et al*. (2022) reported on the chemical modifier to improve properties of coconut shell powder (CSP) filled PLA biocomposites. Their study depicts the effects of filler content on mechanical, thermal and morphology properties as evaluated.

Groundnut (*Arachis hypogaea*) also known as peanut or legume is grown in several countries as a cash crop. The top three groundnut producing nations are China, India, and Nigeria with an annual production of 16,685,915, 6,857,000, and 3,028,571 metric tons, respectively (Mohammed *et.al.,* 2021*;* Maidawa and Dukku, 2022). Groundnut shell (GS), which is an Agro-waste with little or no commercial value, accounts for 20% of the total weight of the pod. Thus, a large amount of groundnut shells is generated globally, paving way for more studies to search for other viable areas of applications. The shells, a by-product of the processing are an abundant agricultural waste material that decomposes slowly in nature (Adhikari *et al.,* 2019*;* Zheng *et al.,* 2013*)*. Despite their potential, GS are often discarded, leading to environmental harm through burning or disposal in landfills. However, these shells contain valuable bioactive compounds and functional ingredients that can benefit various industries, including food, feed, paper, and bioenergy (Trivedi, *et al*. 2019). To achieve zero waste and unlock the full potential of groundnut shells, innovative technologies and applications are necessary to transform this waste into a valuable resource (Rosa *et al.,* 2021*;* Matumba *et al.,* 2017*;* Venkatarajan and Athijayamani, 2020*)*.

Researchers have explored various ways to utilize GS over the years. However, little fraction was successfully incorporated into animal feed, particularly for cattle, while others have investigated their potential as a dietary fiber source for humans. Additionally, studies have examined the use in pulp production, bioethanol production etc. (Yakub *et al.,* 2016). Particle board manufacturing, mulch, and activated charcoal were also investigated (Trivedi, *et al.,* 2019). These efforts aim to find valuable applications for GS. The reduction in waste and unlocking their economic potential are key to engineering advancement. Other applications of GS include its use in composting wet materials, wastewater treatment, plastic, wardrobes, insulation board, metal casting, and a medium for pesticides as well as activated carbon (Berthet *et al.,* 2015). This study investigates the potential of groundnut shell-filled polylactic acid (PLA) composites as a sustainable alternative to conventional petroleum-based materials. Groundnut shells were processed and incorporated as lignocellulosic fillers into a PLA matrix to develop lightweight, eco-friendly composites.

2. Materials and Method

2.1 Sample collection and preparation

Groundnut shells were collected from farm after milling at Kaleri, Bauchi State, Northeastern Nigeria.4 kg of the materials were collected one month after the milling on the farm site. The groundnuts husks were collected and packaged in a bag for this research. The husks were initially dried at 105^{o}C for 24 hours in an industrial oven to remove excess moisture according to the *ASTM D4442*. The PLA grade 2002D was obtained from the Nature Works LLC (USA) IngeoTM grade and was supplied in the form of granules. The properties of the matrix were: density of 1.25g/cm^{3}; glass transition temperature (Tg) of 59^{o}C; melting point of 152^{o}C and melt flow index (MFI) of 30.3g/10 min. Groundnut shell were pulverized into two different mesh sizes of 150 µm and 212 µm using Fritsch Pulverisette P15/P16 mill. The pulverized samples were subjected to 105°C temperature for 24hrs in an industrial oven in order to remove moisture before blending with the polylactic acid granules. Plate 1 depict mesh sizes of GSP.

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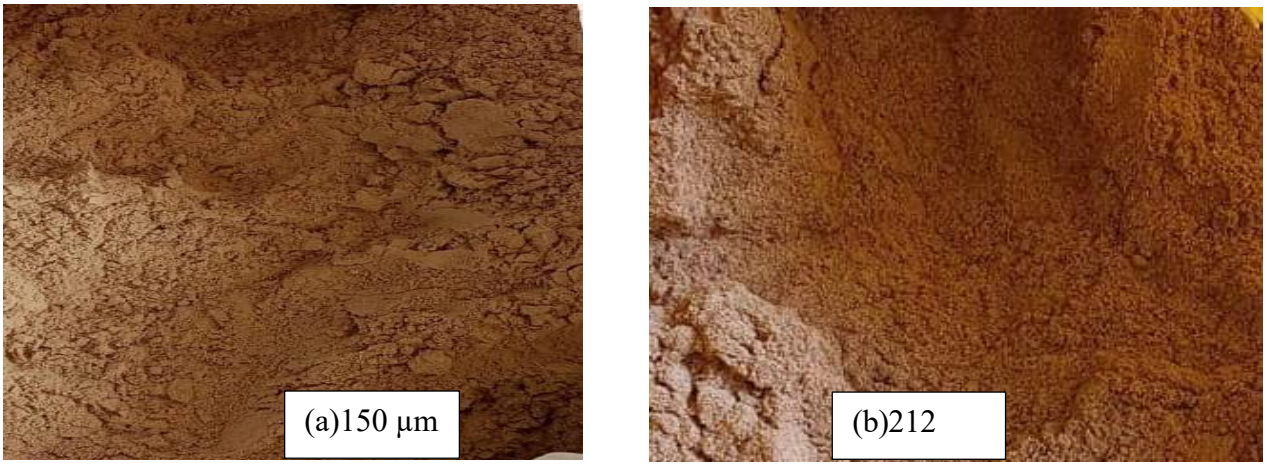


Plate 1(a and b) Mesh sizes of GSP

2.3 Characterization

2.3.1 Chemical composition of the groundnut shells

These analyses were carried out in the Agriculture Chemical Analysis Laboratory (D 10), Food and Agriculture Analysis Laboratory Programme Technical Services Centre, Nigeria. The analyses were performed to evaluate the cellulose, hemicellulose, lignin, and other compounds in the fiber that are mainly responsible for fiber-matrix compatibility in composites. The neutral detergent fiber (NDF) analysis (D10/M1 and D10/M10 In-House-Method) was used to determine the cellulose content of the fiber. The acid detergent fiber (ADF) analysis (D10/M12 and D10/M5 In-House-Method) was employed to determine the hemicelluloses content of the fiber. Meanwhile, the acid detergent lignin (ADL) analysis (D10/M13 and D10/M7 In-House-Method) was used to determine the lignin content of the fiber.

2.3.2 Composites fabrications

Groundnut shell fibers and PLA were compounded by mixing, followed by compression molding process. Internal mixer (Brabender GmbH) was used in the blending process at processing temperature of 170°C, for a period of 10min, and rotating speed of 50rpm. This method was adopted by Collins and Ghodke (2018). labquip scientific 40-ton compression molding machine was used in hot pressing the test specimens. 15 cm ×15 cm × 0.3cm steel mold was used for the compression process. The samples were pre-heated for 3min at 170 °C. Venting time was set to 3min and final press temperature of 170°C for 3min. Finally, the samples were cold pressed for 5min at room temperature. GSP-PLA composites were cut from the two sample sizes with 0%, 10%, 20% and 30% weight of fiber loading.

2.3.3 Composite formulation

Rule of mixture (ROM)

This process involves the rules guiding the mixing of fiber and matrix in composite formulations as shown in equation 1 and 2. In this study, the ROM was based on the capacity of the internal mixer (Brabender). The weight of the fiber and matrix was governed by the rule (*Hammajam et al. 2019)*

$$\text{ROM; } W_f + W_m = 1 \quad (1)$$

$$W_{mf} + W_{hdpe} = M_{cw} \quad (2)$$

where W_f is weight of fiber, W_m is weight of matrix, W_{mf} is weight of groundnut shell powder, W_{pla} is weight of Polylactic acid, M_{cw} is mixer capacity. Table 1 shows the compression conditions for the composite formulation.

Table1: Formulation of GSP-PLA

Sample Name	GSP (wt %)	PLA (wt %)
Neat PLA	0	0
GSP- PLA	10, 20, 30	90, 80, 70

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2.3.4 Mechanical properties

The tensile properties were tested according to *ASTM D638* by using Instron 3365 machine. Tensile testing was carried out at 5mm/min with load cell of 5kN. While the flexural properties were tested by using the same machine that was used for the tensile test at a crosshead speed of 3 mm/min with a load cell of 5 kN and a span of 50 mm. Hardness test is extensively used to characterize a certain material and to identify if it is appropriate for its intended purpose. Hardness test is the measure of its resistance to the indentation due to compression force caused by sharp tools. Durometer hardness test is carried out based on the *ASTM: D4762* identify the hardness of the natural fiber composite material. A similar method and process was reported by Hammajam, et al. (2019) and Sapuan and Bachtiar (2012). Plate 2 shows the mechanical properties test specimens. While Figures 1 and 2 shows flexural and tensile test specimen as per *ASTM D790; D638* respectively.



Plate 2: Mechanical properties test specimens

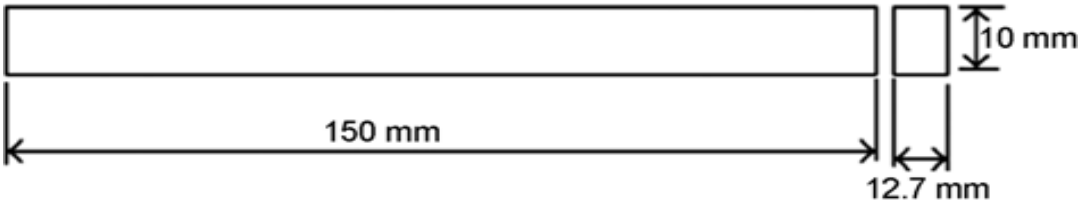


Figure 1: Flexural test specimen (ASTM. D790)

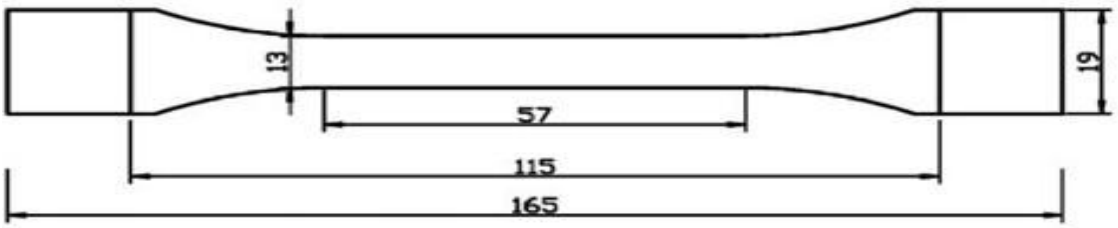


Figure 2: Tensile test specimen as per ASTM D638

2.3.5 Microstructural examinations

The fiber-matrix dispersion analyses were carried out to understand the extent of fracture surface when the composites were subjected to tensile testing. The fiber dispersions were inspected using Scanning Electron Microscope (SEM). This process was done by using Hitachi S–3400 N model at 20.0 kV.

3. Results and Discussion

3.1 Chemical composition analysis of the GSP

The chemical compositions of GSP are shown in Table 2. It was observed that the cellulose content of the GSP was 43.2%. This value was different from the 32.7% and 33.2% values reported by Patel, (2012) and

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Petchwattana and Covavisaruch, (2013) respectively. It was also observed that the shell's hemicelluloses and lignin contents were 24.17% and 16.19%, respectively. Whereas It was also revealed that the protein and fat contents were 7.63% and 4.31%. Other constituents of the fiber; pectin, waxy contributed to the remaining percentages (%) of the GSP. The general observation in natural fibers was that the expected moisture uptake in fiber takes place mainly by hemicellulose, starch, lignin and surface of cellulose content. Lignocellulosic cereal fiber contains fat and protein which are covalently bonded together, and act as a fiber coating. Hence, there are slight variations in the composition of cereal byproducts, as has been reported by *Andrzej et al. (2010*).

Table 2: Chemical composition of the groundnut shells

Composition	Cellulose	Hemicellulose	Lignin	Protein	Fat
% content	43.2	24.17	16.19	7.63	4.31

3.2 Tensile Strength of GSP-PLA Composite

Figure 3. depicts the mean values of tensile strengths of GSP–PLA composites with different fiber loading. in composites development, there is a complicated interaction of the dynamics of the basic stages such as matrix, fiber and interfacial zone between the fiber and matrix as reported by Ridzuan, et al. (2016). The increase at 10 % by weight of GSP causes slight decrease in the tensile strength of GSP–PLA composites when compare to control sample (0 % PLA. It was observed that composites with 20 % fiber loadings appreciated in strength of 14.2 MPa and 15 MPa for 150 µm and 212 µm respectively. Thus, it shows slight decrease below 0 % PLA with 25.9MPa. Furthermore, it can be said that fiber size has effect on the tensile strength of GSP-PLA composites. This shows similar finding in Kanayo, et al. (2018). Hence the decrease in the tensile strength is due to poor GSP–PLA interaction as a result of opposing separations of the GSP and PLA matrix. Also, in Figure 1, increase loading up to 30 % has slightly decreased the tensile strength of the composite for both the fiber sizes. This could be due to weak dispersion and weak interface in the PLA matrix resulting in poor bonding between GSP and PLA.

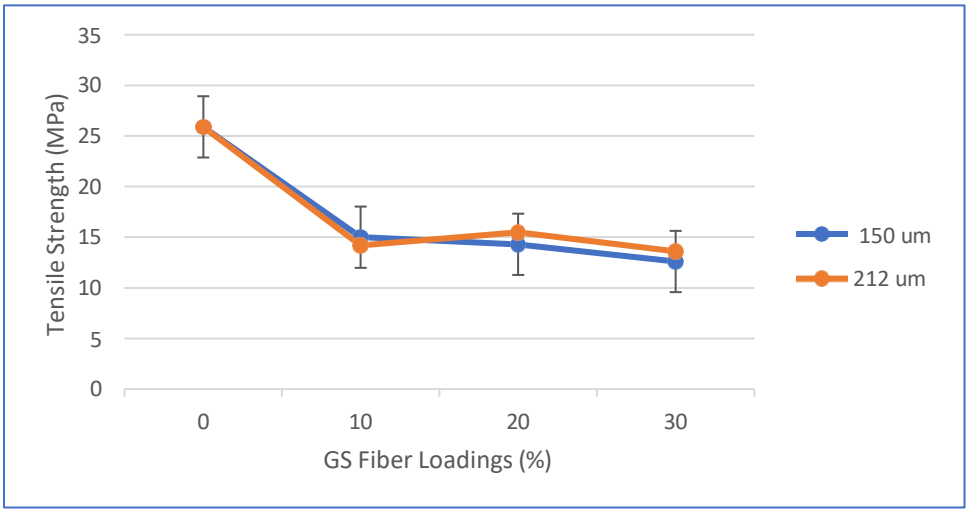


Figure 3: Effect of fiber loading on the tensile strength of GSP-PLA Composite

3.3 Tensile Modulus of GSP-PLA Composite

From Figure 3, there was an increase in the tensile modulus of GSP–PLA composites as fiber loadings increased. It can be observed that the maximum value of the tensile modulus was 712 GPa at 10 % loading for 150 µm. it was drop to 472 GPa at the loading for 212 µm. at 20% loading, the modulus increases for 212 µm and decrease for 150 µm. But at 30 % loadings, 150 µm continue to decrease while 212 µm appreciated. Thus, it was still appropriate to argue that increasing GSP up to 30 % in PLA increases the rigidity of the composites. It showed the level of adhesion between GSP-PLA hardly affect the tensile modulus significantly. The tensile moduli of the composites are enlarged by increasing the fiber loadings. The reason is the fact that lignocellulosic fibers possess greater modulus when compared to polymer matrix. Also, because modulus is processes that involve so small strain values and small stress may be produced with application of little value of elongation (Mohammed, *et al*. 2021). Thus, it was expected that there will be further improvement of modulus of the composites when going above 30% fiber loadings for 212 µm.

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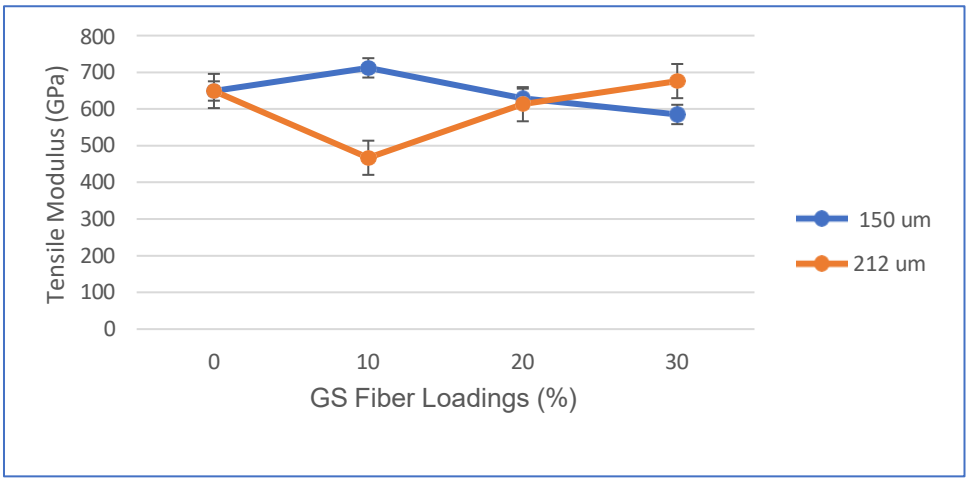


Figure 4: Effect of fiber loading on the tensile modulus of GSP-PLA Composite

3.4 Percentage Elongation of GSP-PLA Composite

It was observed from figure 5 that the tensile elongation at the break of the composites was grossly decreased. This is the result of thermoplastic composites having a rigidity tendency because of the increase in fiber loading. Usually, the reduced tensile elongation at break values is as a result of increased modulus. Similar findings were reported by Mohammed *et al*. (2021). It is possible that the addition of fibers which has brittle attribute decrease the elongation at break.

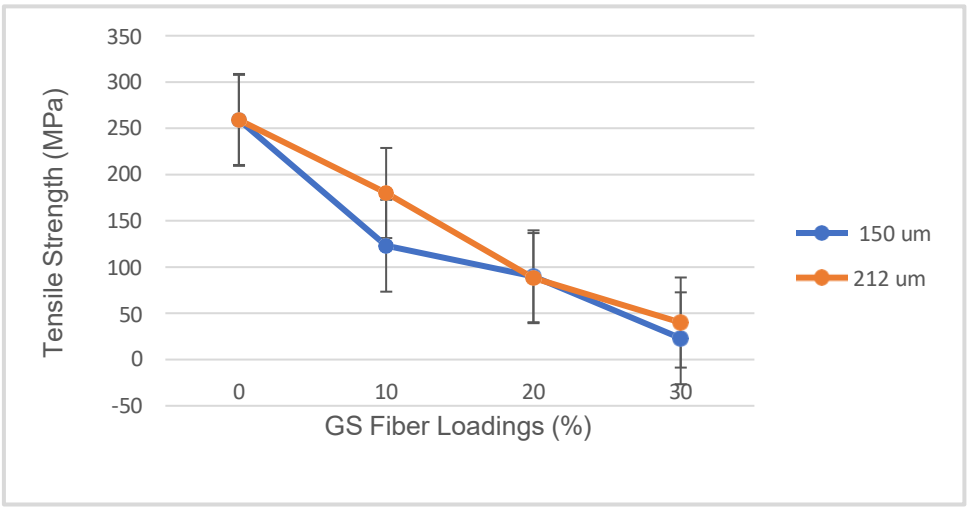


Figure 5: Percentage Elongation of GSP-PLA Composite

3.5 Flexural strength of GSH-PLA Composite

Figure 6 depict the effects of fiber loadings on the flexural properties of the GSP-PLA composites. The flexural strength of the GSP-PLA composites increased for the 10% and 20% fiber loadings for 150 µm but decreased at the 20% fiber loadings for 212 µm. There was an increase as fiber loadings reached 30% for 150 µm and dropped slightly for 212 µm when compared to pure PLA. It was observed that the flexural strength dropped 11.9% at 20% fiber loading for 150 µm. The findings show an increase in strength at 10% and 20% fiber loadings for PLA composites. Therefore, the fiber did not reduce completely the ability of fibers and matrix to interact. The increased adhesions create better fiber and matrix interactions which allow the stress to be transferred from the matrix to fiber and translates into improved flexurability. Similar findings were reported by Yakub *et al*. (2017). The increase in flexural strength became highly prominent when the fiber loading did not go beyond 10% and 20%. Furthermore, this is only likely when there was a better stress transfer from matrix to fiber via a good interface adhesion.

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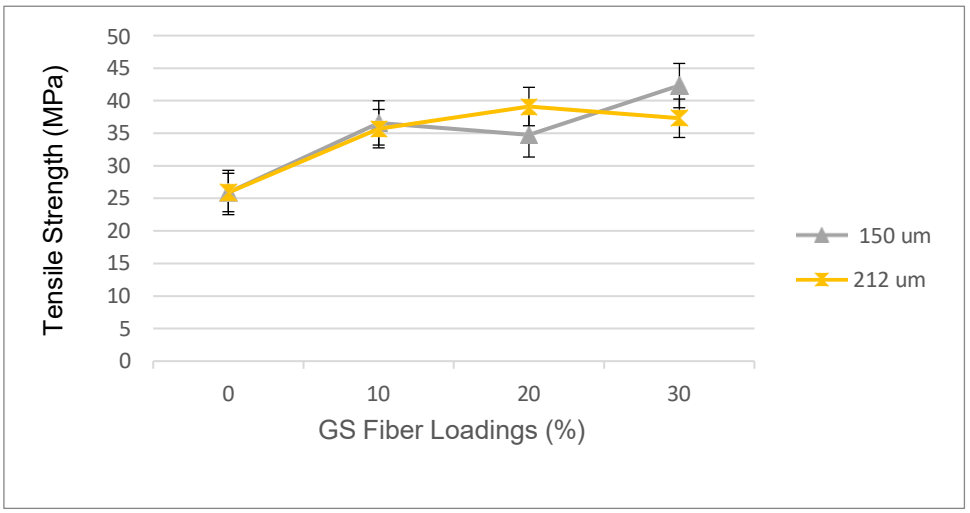


Figure 6: Flexural strength of GSP-PLA Composite

3.6 Flexural Modulus of GSP-PLA Composite

In most cases, the normal trend for flexural modulus is to increase with increasing fiber loading. These characteristics were indeed observed in Figure 7. this could be attributed to the contribution made by fibers to impart their own property to the composite. The findings reveal a steady increase in the modulus values as the fiber loading is increased from 712.9GPa for pure PLA to 2344.9 GPa at 30% loading in 150 µm and 1576 GPa for 212 µm. It was revealed that there was around 45% improvement in flexural modulus up to 30% fiber loading from neat PLA and 44.7% from 10% fiber loading for the PLA composites. These phenomena presume that above 30% fiber loading, the composites stiffness will decrease as the fiber loading increases. Therefore, the increase in the modulus of these composites was due to the higher initial modulus of the fibers acting at the main building block of composites and improve interfacial bond between the fibers and matrices

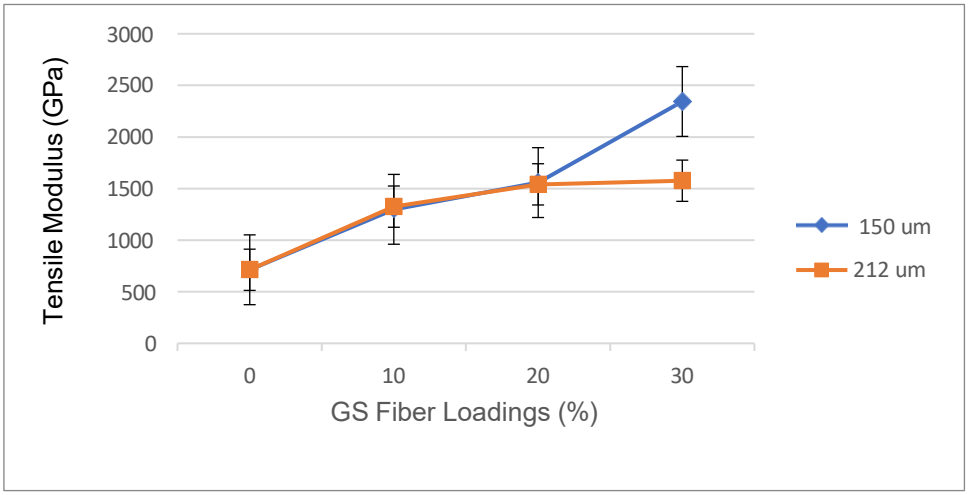


Figure 7: Flexural Modulus of GSP-PLA Composite

3.7 Result of Hardness Test of GSP-PLA Composite

Hardness of a material is the measurement of its resistance to dentation due to a compressive force caused by a sharp object. The results of hardness test are shown figure 8. that hardness of GSP-PLA There is increased in hardness throughout the loadings. Although, at 20% loading, is slightly decreased. It also revealed that angle orientation has play much role in the hardness property of the composite. This is because as the fibre loadings increases, the resistance to penetration is more due to the availability of fibres acting as rigid medium.

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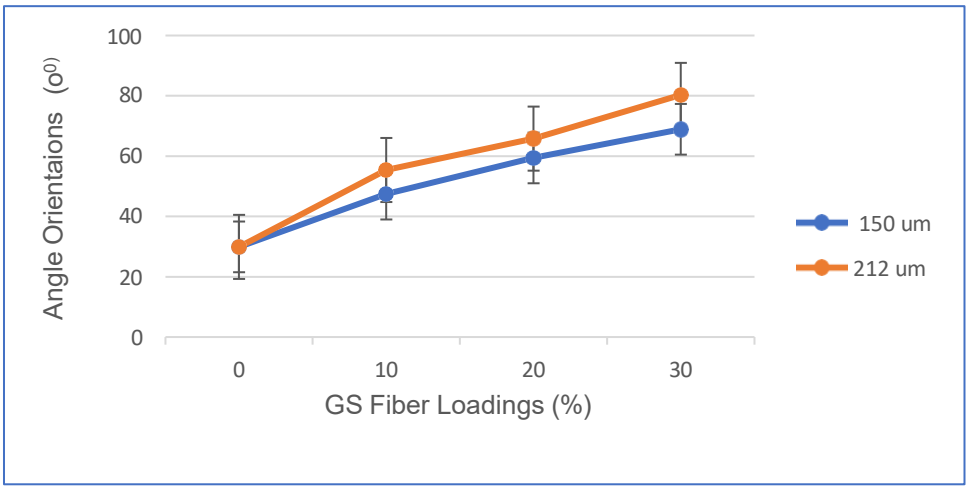


Figure 8: Hardness Test of GSP-PLA Composite

3.8 Microstructures Observation of GSP-PLA Composite

SEM images of fractured surface of GSP–PLA composites were depicted in plates 3(a and b). The image of different fiber loading composites shows that lower GSP loadings indicates non–homogeneous dispersion patterns. This causes fiber pull–out and makes the fiber to undergo accumulation. Hence, these lead to reduction in the tensile strength of the composites mostly at lower fiber loadings. Similar findings were reported by Fogorasi, and Barbu, (2017). However, at 20 % and 30 % loadings, there were better dispersion of GSP–PLA formation with reduced fiber pull out and breakage. This is the main reason for improvement of tensile strength at these loadings for smaller fiber sizes.

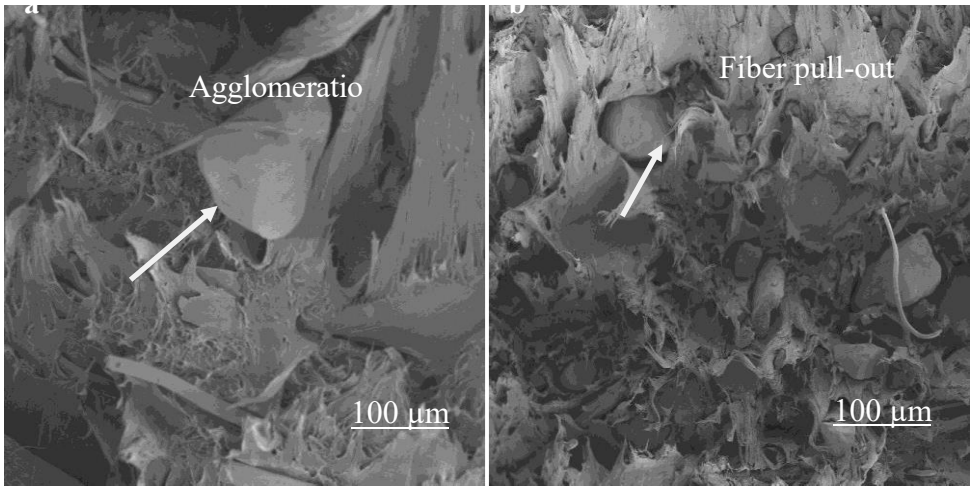


Plate 3a: SEM micrographs 150 µm of GHP-PLA composites

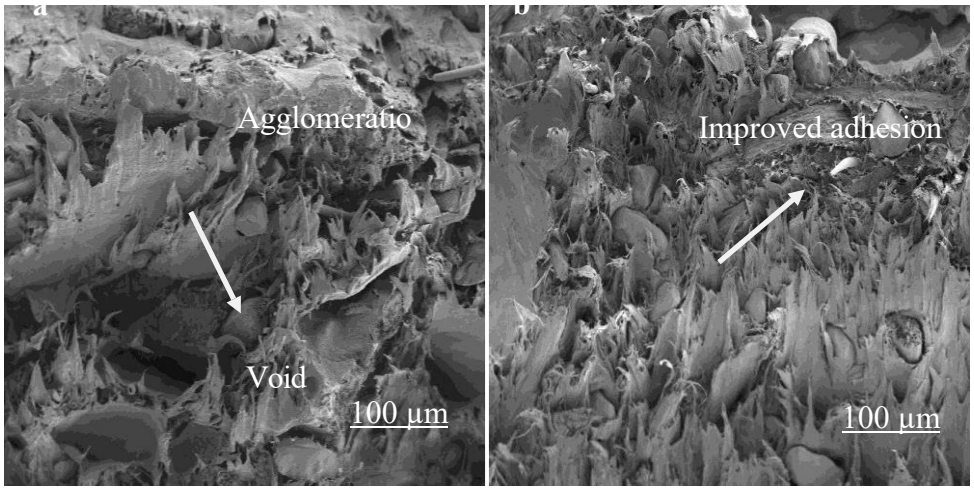


Plate 3b: SEM micrographs 212 µm of GHP-PLA composites

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4. Conclusion

This study had shown that fiber loading significantly influences its mechanical properties. It was observed that composites with 20 % fiber loadings appreciated in tensile strength of 14.2 MPa and 15 MPa for 150 µm and 212 µm respectively. Interestingly, it was observed that there was a steady increase in the tensile modulus values as the fiber loading is increased from 712.9GPa for pure PLA to 2344.9GPa at 30% loading in 150 µm and 1576 GPa for 212 µm. The optimum performance strength for both composites type was at 20% filler loading and the modulus was at 30% filler loading GSP-PLA composite. Consequently, the flexural strength of the GSP-PLA composites increased for the 10% and 20% fiber loadings for 150 µm but decreased at the 20% fiber loadings for 212 µm. It was revealed that there was around 45% improvement in flexural modulus up to 30% fiber loading from neat PLA and 44.7% from 10% fiber loading for the PLA composites. Thus, from these findings, the developed composites possess attributes that suit some engineering applications such as automobile interiors and household utensils were high impact are less needed.

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