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M.F.A. HASHIM^{01,2*}, Y.M. DAUD^{01,3}, M.M. AL BAKRI ABDULLAH^{01,3}, M.A. FARIS^{01,2}, M.S.M. RASIDI^{01,3}, C.M.R. GHAZALI^{01,4}, F.F. ZAINAL^{01,3}, S. HASYIM⁵, N.N.M. NAZRI², S. GARUS⁶

EFFECTS OF DIFFERENT FIBER SIZES IN PLA/CARBON FIBER COMPOSITES **ON MECHANICAL PROPERTIES**

This study assessed the morphology and chemical composition of coir coconut husk carbon fiber, as well as the impact of fiber diameters on the physical and mechanical properties of polylactic acid composites. Researchers are studying polylactide acid, a biodegradable material. This eco-friendly material's excellent features, generated from sustainable and renewable sources, have drawn many people. Malaysia's high coconut fiber output made coir husk a popular commodity. Coconut fibers are lignin, cellulose, and hemicellulose. Alkaline treatment eliminates hemicellulose, oil, wax, and other contaminants from coir fibers and removes lignin. Fourier Transform Infrared Spectroscopy and Scanning Electron Microscopy were used to examine the treated coconut fibers' chemical modification analysis and morphology. Coconut coir husk was carbonized to produce carbon fiber using a furnace operated at 300°C for 2 hours. Fiber and polylactic acid were mixed in different fiber sizes (0, 53 µm, 75 µm, and 212 µm) via extrusion and injection processing techniques. The results showed that the alkali treatment reduced the hydroxyl (-OH) group and separated the area from the carbonyl (C=O) group of coconut coir husk, which changed the filler's hydrophilicity. The fiber size of 212 µm was discovered to have the highest tensile and flexural strength values. According to testing, the modified material structure had a better surface fill-matrix bond. Thus, generalized fiber sizing and characterization methods were developed. Regardless of the matrix, this method can characterize natural fiber strength and interfacial shear strength of varied diameters and solid contents.

Keyword: Carbon fiber reinforced PLA (CF-PLA); Natural Fiber; Coir Coconut Husk; Tensile Mechanical Properties; Fiber-Matrix Interface

1. Introduction

In recent years, there has been a notable emphasis within the polymer sector on the development and utilization of polymers derived from renewable sources. The exploration of natural fiber composites as a potential substitute for conventional synthetic composites has garnered significant attention across various technical disciplines. The suitability of natural fiber composites for fostering affluent and sustainable societies is attributed to their inherent properties. Polylactic acid (PLA) is among the thermosets and thermoplastic matrices typically associated with natural fibers. In order to derive advantages from the applications, each group possesses distinct and unique qualities. Thermoplastic materials provide notable attributes such as high impact resistance and ductility [1].

In recent times, there has been a surge in interest surrounding lignocellulose products. The fundamental impetus behind this endeavour is the necessity to explore alternative raw materials derived from renewable resources. Lignin, a widely abundant natural polymer, is frequently obtained as a byproduct from various pulping procedures and afterwards utilized for on-site combustion to generate heat, owing to its substantial energy density. There are two main categories of fibers: man-made synthetic fibers and natural fibers. The combination of synthesis and renewable materials synergistically contributes to the development of a highly effective structural material that exhibits characteristics akin to those of natural fibers, thereby offering a viable alternative to costly, non-renewable synthetic fibers [2].

When compared to other natural fibers, coconut fiber exhibits several advantages, such as a notable failure strain and

Corresponding author: firdaushashim@unimap.edu.my



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UNIVERSITI MALAYSIA PERLIS, CENTER OF EXCELLENCE GEOPOLYMER & GREEN TECHNOLOGY (CEGEOGTECH), 01000, PERLIS, MALAYSIA

UNIVERSITI MALAYSIA PERLIS, (UNIMAP), FACULTY OF MECHANICAL ENGINEERING AND TECHNOLOGY, PERLIS, MALAYSIA

UNIVERSITI MALAYSIA PERLIS, (UNIMAP), FACULTY OF CHEMICAL ENGINEERING AND TECHNOLOGY, 02600 JALAN KANGAR-ARAU, PERLIS, MALAYSIA

UNIVERSITI MALAYSIA TERENGGANU, FACULTY OF OCEAN ENGINEERING TECHNOLOGY AND INFORMATIC, 21030 KUALA NERUS, TERENGGANU DARUL IMAN, MALAYSIA SRIWIJAYA UNIVERSITY FACULTY OF ENGINEERING, CIVIL ENGINEERING DEPARTMENT, INDORUSIA CZĘSTOCHOWA UNIVERSITY OF TECHNOLOGY, FACULTY OF MECHANICAL ENGINEERING AND COMPUTER SCIENCE, 42-201 CZĘSTOCHOWA, POLAND

enhanced weather resistance attributed to the presence of lignin. The reduced cellulose content in coconut fiber results in a diminished water absorption capacity. Similar to other types of natural fibers, coconut fiber has demonstrated limited effectiveness as a reinforcement material due to its wide and inconsistent diameter, elevated micro-fibril angle, and significant presence of lignin and hemicellulose. Coconut husk, which is the fibrous outer layer of the fruit of coconut palms, is commonly considered waste and is obtained as a by-product during the extraction of copra. Lignin, comprising around 33% of the total weight of the coconut husk, has been proposed to serve various functions [3].

Poly(lactic acid) or polylactide (PLA) is a very promising biomaterial that exhibits great potential for many medicinal applications, offering a viable alternative to standard petrochemical-based polymers commonly employed in industrial settings. The utilization of polylactic acid (PLA), a thermoplastic polymer with notable strength and modulus properties, holds the potential for enhancing the market of biocompatible/bioabsorbable medical devices and industrial packaging. PLA may be derived from annually renewable resources, further contributing to its appeal in both sectors. One of the most extensively researched and utilized biodegradable and renewable aliphatic polyesters is capable of being processed on standard plastic equipment to manufacture molded components, films, or fibers [4].

In recent years, the development of advanced materials has paved the way for numerous breakthroughs in various industries. Among these materials, PLA/carbon fiber composites have gained significant attention due to their exceptional mechanical properties. However, the specific influence of different fiber sizes on the mechanical properties of these composites remains relatively unexplored. This research aims to address this research gap by investigating the effects of different fiber sizes in PLA/carbon fiber composites on their mechanical properties. By evaluating the impact of fiber size on the overall performance and strength of these composites, this study aims to shed light on the critical role of fiber size in enhancing their mechanical properties. Through a comprehensive analysis of the experimental results, this investigation reveals promising findings that solidify the significance of fiber size as a crucial factor in optimizing the performance of PLA/carbon fiber composites.

2. Experimental

2.1. Materials selection

The primary material utilized in this investigation is polylactic acid (PLA), which was procured from TT Biotechnologies Sdn. Bhd., located in Penang. The PLA polymer is sourced from renewable natural resources, including wheat, corn, and sweet potato. Polylactic acid (PLA) is a biodegradable and renewable polymer that has gained significant attention recently due to its potential applications in composite industries. According to Siakeng et al. (2019), PLA possesses numerous advantageous properties, such as high mechanical strength, good thermal stability, and excellent processability, making it an attractive alternative to conventional petroleum-based polymers. The authors further argue that PLA can be blended with other reinforcing materials, such as natural fibers, to enhance its mechanical properties and create sustainable composites with a lower environmental impact. This aligns with the growing demand for eco-friendly materials in various industries. Furthermore, the potential of PLA in composite industries is not limited to its mechanical properties. Siakeng et al. (2019) also highlight the potential for functionalizing PLA composites by incorporating nanoparticles or conducting polymers, which could open up new opportunities for applications in electronics, aerospace, and other highperformance industries [5].

The second primary material utilized in this study is the coconut coir husk (CCH), which was procured from MAAS Agro Technologies. The research utilizes dried coconut coir husk fiber, as depicted in Fig. 1. The fiber composition of CCH consists of cellulose, hemicellulose, and lignin.



Fig. 1. Dried Coconut Coir Husk Fiber

In this study, the chemical alteration of CCH fibers was conducted using sodium hydroxide (NaOH), ethanol (CH₃CH₂OH), and sulfuric acid (H₂SO₄). The sodium hydroxide (NaOH) was acquired in flake form from AGC Chemicals (Thailand) Co. Ltd., while the ethanol and sulfuric acid were provided by A. R. Alatan Sains (K) Sdn. Bhd.

2.2. Sample preparation

2.2.1. Coconut Coir Husk Preparation

Initially, the CCH (calcium carbonate hydrate) is going through a drying process within a controlled environment, namely a drying oven, at a temperature of 60°C for a duration of 24 hours. This procedure is employed to eliminate any residual moisture content present within the material. Subsequently, the specimens are subjected to grinding using a desktop high-speed grinder (Model: RT-34) with a power output of 1 horsepower (HP). The ground CCH was subsequently stored in a container for the purpose of identifying its functional groups and doing additional processing.

Properties of Coconut Coir Fibers

TABLE 1

Properties	Coir
Density (g/cm ³)	1.2
Modulus (GPa)	4-6
Tensile Strength (MPa)	175
Elongation to failure (%)	30
Water absorption (%)	130-180

2.2.2. Chemical modification

The chemical alteration of ground coconut coir husk involves using an alkali, specifically Sodium Hydroxide (NaOH), in conjunction with 99% ethanol (CH₃CH₂OH). The alkaline solution was made by thoroughly combining 2.5% sodium hydroxide (NaOH) with distilled water and ensuring proper agitation.

The ethanol was diluted in a 1:1 ratio with distilled water and thereafter introduced into the NaOH solution. Subsequently, the amalgamation was united with CCH and subjected to a temperature of 170°C for a duration of 1.5 hours within the reflux apparatus arrangement, as seen in Fig. 2. Following the application of heat to the CCH, it underwent a rinsing process, including a 2% H_2SO_4 solution that had been appropriately diluted with distilled water in order to restore the pH of the treated CCH to a neutral value of 7. The CCH was thereafter washed with distilled water until the absence of any discernible odour from the solution could be perceived. Subsequently, the CCH fiber that



Fig. 2. Variable Soxhlet Extraction Apparatus

had undergone treatment was subjected to a drying process at a temperature of 70°C for a duration of 6 hours until the weight of the CCH achieved a state of equilibrium. The dried and processed CCH was thereafter placed in a hermetically sealed container for the purpose of conducting functional group identification tests using Fourier Transform Infrared Spectroscopy (FTIR), as well as for subsequent processing.

2.2.3. Carbonization process of coconut coir husk

The fibers undergo a carbonization process, wherein the PAN-based fibers undergo a transformation into carbon fibers by eliminating oxygen, hydrogen, and nitrogen from the precursor fiber. The process of carbonization is employed due to its ability to enhance elasticity while compromising strength through the phenomenon of graphitization. The process of carbonization was accomplished by subjecting the material to a gradual heating rate of 5°C per minute until it reached a temperature of 300°C. It was then allowed to soak at this temperature for a duration of 1 hour. Subsequently, the material was cooled down at the same rate of 5°C per minute until it reached room temperature, resulting in a total carbonization time of 11.8 hours. In an alternative approach, the precursor fibers (PFs) underwent carbonization under identical conditions, omitting the oxidative stabilization process. This subsequent method would be referred to as immediate carbonization. The carbonized CCH fiber was further pulverized using a mortar and pestle and then filtered through a screen with a mesh size of 75 µm. The carbon material, which had undergone sieving, was afterwards placed in a container that was tightly sealed in order to facilitate subsequent processing.

2.2.4. Formulation of material

In order to examine the impact of varying fiber sizes in composites, the present study employed the parameter outlined in TABLE 2. The quantities of PLA pellets and carbon CCH fibers were determined in accordance with the values provided in TABLE 2.

TABLE	2
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Mixing	Ratio
1.	1.0001.0

Mixing Coconut Husk Carbon Fiber (CCH) with Polylactic Acid (PLA)					
Sample	Control	Α	В	С	
CCH Size (mm)	0	53	75	212	
Fiber Loading (g)	0	22.5	22.5	22.5	
PLA (g)	150	127.5	127.5	127.5	

2.2.5. Extrusion molding process

The temperature of the extrusion machine was adjusted to 165°C while the screw speed was set at 15 rpm. A pellet of

polylactic acid (PLA) was combined with carbon CCH fiber and introduced into the extrusion hopper for the purpose of compounding. The composite material was extruded in the shape of a string and afterward introduced into a pelletizer for the purpose of cutting it into pellets of the desired size. The PLA/ carbon CCH composite pellet underwent a drying process in an oven at a temperature of 40°C for a duration of 4 hours prior to proceeding with the subsequent steps.

2.2.6. Injection molding process

Carbon fiber pellets made from PLA/CCH will be put into the feeding hopper of the injection machine. The operational temperature of the machine will be adjusted to 165°C in order to facilitate the melting of the pellet. The molten composite material will be introduced into a mold by an injection process. The composite product will be allowed to solidify before its removal from the material.

2.3. Physical and mechanical characterization

Several physical and mechanical tests have been done in this study: density test, water absorption test, functional group characterization, tensile strength test, flexural strength test, and morphology characterization to determine the properties of the effect of different fiber sizes in PLA/CCH fiber composites.

2.3.1. Density test

The density test will utilize two distinct testing methods: a manual test and the utilization of densimeter equipment that has been previously used for testing in water. In the context of densimeter machines, a specific methodology will be employed to acquire the specific gravity (SG) value. Subsequently, the average SG value will be calculated utilizing the provided equation.

$$\rho = \frac{m}{v} \tag{1}$$

Where *m* is the mass of the neat PLA/carbon fiber while *v* is the volume of the neat PLA/carbon fiber.

2.3.2. Water absorption test

The water absorption test employed ASTM D570 as the standard method for quantifying the proportion of water absorbed. The study employed composite samples, as depicted in Fig. 3, which possessed a uniform thickness of 3 mm. The samples were cut using a saw, and subsequently, the edges were meticulously sanded with 1200 Grit sandpaper to ensure a flaw-less surface without any flaws. Subsequently, the specimens were subjected to a drying process in an oven set at a temperature

of 40°C for a duration of 24 hours. Following the completion of the drying process, the specimens were promptly weighed with precision to the nearest 0.001 g. The specimens were fully submerged in a vessel containing distilled water and kept at a controlled ambient temperature of $23 \pm 1^{\circ}$ C for a duration of 24 hours. Following a 24 hours period, the specimens were individually extracted from the water, ensuring that all surface water was eliminated by means of a dry cloth. Subsequently, the specimens were promptly weighed to the nearest 0.001 g.

2/1/23 12.25 102 2/1/23 12.25 102 2/1/23 12.25 12.25 12.25 12.25 12.25 12.25

Fig. 3. Submerged PLA/Carbon CCH Composite Samples for Water Absorption Test

2.3.3. Tensile strength test

Fig. 4 demonstrates the experimental procedure for conducting a tensile strength test using a SHIMADZU Universal Test



Fig. 4. The Illustration of The Tensile Strength Test

Machine (UTM) with a maximum load capacity of 50kN. The velocity of the crosshead was adjusted to 3 mm/min. The specimen was employed in accordance with the ASTM D638 standard. The observed shape exhibited a configuration resembling that of a dumbbell. The thickness, width, and length of the specimens were measured, and their gauge lines were appropriately labeled. The specimen was subjected to a tensile testing procedure, during which a load was gradually added until the specimen reached its breaking point. Determining tensile strength involved identifying the peak force from the force-displacement curve.

2.3.4. Flexural strength test

Fig. 5 illustrates the flexural strength test conducted according to ASTM D790-17. The flexural test is commonly utilized to analyze the mechanical properties of composite materials. The experiment involved subjecting a rod specimen with either a circular or rectangular cross-section to bending until it fractured. This was achieved by employing a three- or four-point loading approach. The experiment involved conducting flexural testing on rectangular bars at a temperature of 23°C. The tests were conducted with a cross-head speed of 1.3 mm/min and a distance of 50 mm between the support spans. The tests were performed until a strain of 5% was reached.



Fig. 5. The Illustration of The Flexural Strength Test

2.3.5. Morphology characterization

In addition to assessing followability and conducting mechanical tests, the microscale fracture pattern of each fiber type was examined using a Scanning Electron Microscope (SEM). Multiple samples were extracted from the fracture plane subsequent to mechanical testing of chopped mixes (CH2), milled mixes (M2), and hybrid mixes (H2). Using a TESCAN (Brno, Czech Republic) VEGA3 thermionic emission SEM system, the fracture surfaces of the specimens were analyzed. In order to improve conductivity, the specimens underwent a process of sputter-coating with a gold/palladium (Au/Pd) compound. The energy of the electron beam employed for all measurements was 20 kilo electron volts (keV). The degree of magnification ranged from $400 \times$ to $1150 \times$, contingent upon the specific features under observation.

2.3.6. Functional group characterization

Samples of fibers from both untreated and treated CCH and carbon CCH were collected for the purpose of identifying the functional group of the material. This was achieved by the use of Fourier Transform Infrared-Spectroscopy (FTIR) using a Perkin Elmer Spectrometer Frontier. The obtained data was subsequently analyzed using Perkin Elmer Spectrum Version 10.5.2. The samples were combined with potassium bromide in a ratio of 1:2. The powder mixture was thereafter subjected to compaction for a duration of 2 minutes, utilizing a force of 8,000 kg. The spectral range spanned from 4,000 cm⁻¹ to 650 cm⁻¹.

3. Results and discussion

3.1. Functional group analysis

Fig. 6 displays the infrared (IR) spectra of both untreated and treated CCH. Fourier Transform Infrared Spectroscopy (FTIR) was conducted in order to analyze and monitor the chemical alteration of the treated Cellulose Chitosan Hybrid (CCH). The graph displays the presence of hydroxyl (O-H) stretching in both the untreated and treated samples, with corresponding peaks at 3366 and 3252 cm⁻¹. Nevertheless, the untreated graph has a more pronounced and smaller infrared (IR) band associated with the O-H group. In the graph depicting the treated CCH, it is evident that the O-H group band exhibits a diminished intensity and a larger infrared (IR) band. This observation suggests that the hydrogen bond present in the CCH was disrupted after the alkali treatment [6]. The methylene (C-H) stretching of an alkane is observed at wavenumbers 2920 and 2921 cm⁻¹. In its untreated state, the stretching is characterized by a stronger and narrower band. However, after treatment, the stretching exhibits a reduced intensity and a broader infrared (IR) band. Alkali treatment of coconut coir husk has been found to have several effects on the mechanical properties of the resulting fibers. The treatment increases the surface roughness of the fibers, which



Fig. 6. FTIR Spectrum of Raw Coconut Coir Husk and Treated Coconut Coir Husk

728

can enhance their adhesion to a matrix material. Additionally, alkali treatment has been shown to reduce the amount of hemicellulose in the fibers, while increasing the percentage of cellulose and lignin. This can lead to improved interfacial bonding with a matrix material, ultimately enhancing the mechanical properties of the composite [7].

3.2. Density analysis

Fig. 7 illustrates the relationship between density measurement and the size of carbon fiber coconut husk reinforcement in polylactic acid (PLA). The density of the PLA/carbon CCH fiber, characterized by a fiber size of 0 μ m and a carbon fiber content of 0%, was measured to be 1.236 g/cm³. The graph in Fig. 7 also illustrates a relationship between the size of carbon fiber and its corresponding density. It can be observed that as the size of carbon fiber increases, there is a corresponding increase in density. For instance, carbon fibers with a size of 53 μ m exhibit a density of 1.250 g/cm³, whereas carbon fibers with a size of 75 mm demonstrates a significant decrease in density to 1.194 g/cm³. Based on the data presented in the graph, it can be observed that the average density of carbon fiber with a fiber size of 212 μ m closely approximated that of carbon fiber with a fiber size of 53 μ m, measuring at around 1.249 g/cm³.



Fig. 7. Density (g/cm³) of Pure PLA and PLA/Carbon CCH Composite with Different Fiber Size

The presence of carbon CCH fiber in the sample, resulting from the dispersion of fibers during the extrusion process, might lead to variations in density and mass. The uniform distribution of carbon CCH fiber throughout the sample was affected by the displacement that took place during the extrusion process, hence influencing the properties of the sample.

As shown in Fig. 7 earlier, it is evident that the sample density for the two procedures exhibits a substantial difference only at a carbon CCH fiber thickness of 75 μ m. As a result, there was an increase in fiber loading that exceeded initial expectations. This phenomenon exhibits a strong resemblance to the prior investigation conducted by Teymoorzadeh and Rodrigue (2016) [8]. The presence of carbon fibers (CFs) in the PLA

matrix is anticipated to disrupt the crystallinity, whereas the quantity of gas absorbed in the composites is contingent upon the fiber loading inside the composite system [9]. The high fiber loadings in PLA/KF composite foams have a significant impact on the interaction between the fiber and matrix. This is mostly due to the insufficient wetting of the fiber by the polymer matrix. Consequently, the density trend of these composite foam exhibits fluctuations [10,11]. The density of the composite can be influenced by various factors such as the infill density, fiber content, and manufacturing parameters. For instance, increasing the carbon fiber content in the composite can lead to a change in density. Additionally, the infill density has been found to influence the mechanical and thermal properties of the composites, with studies reporting that an increase in infill density can enhance tensile strength and fatigue life [12-14].

3.3. Water absorption analysis

In order to ascertain the quantity of water absorbed under specified circumstances, a water absorption test was carried out, as seen in Fig. 8.



Fig. 8. Water Absorption (%) of Pure PLA and PLA/Carbon CCH Composite with Different Fiber Size

Fig. 8 illustrates the water absorption data pertaining to the incorporation of carbon coconut coir husk (CCH) fibers in polylactic acid (PLA). Following a 24-hour immersion period in distilled water, the carbon CCH fiber exhibits a water absorption rate of 0.20% for pure polylactic acid (PLA). The water absorption capacity of fibers with a size of 53 μ m showed a growth rate of 0.3224%. However, there was an increase in fiber size for 75 μ m, contributing 0.3945%. According to the data presented in the graph, it is evident that the fiber with a diameter of 212 μ m exhibits the highest rate of water absorption, measuring at 0.4178%. Consequently, water absorption increases in the presence of various particles of varying sizes.

The graph illustrates a substantial disparity in weight that may transpire during a 24-hour timeframe. The weight change of the sample was found to be below 5%. This weight change can be attributed to the exposure of the sample's fibers to water, resulting from the alteration in the sample's cross-section. As evidenced by the graph, the composite sample exhibits a higher water absorption capacity compared to pure PLA. In contrast, a significant portion of the sample weight exhibits an increase. The reason for this phenomenon can be attributed to the thorough blending of coconut fiber with polylactic acid, resulting in an expression of water absorption as a percentage rise in weight. The observed enhancement in water absorption rate can be attributed to the incorporation of carbon fibers (CFs), which resulted in an increase in the overall mass of the foam composites consisting of polylactic acid (PLA) and CFs [15]. This is proven as, in conjunction with a study reviewed by Alawar, Hamed, and Al-Kaabi (2009), the effects of varying fiber sizes on water absorption capacity were investigated. The results indicated that the water absorption capacity of the composites increased as the fiber size decreased. This finding can be attributed to the fact that smaller fiber sizes provide a larger surface area for water molecules to interact with, leading to increased water absorption. Additionally, the researchers noted that the interfacial bonding between the fibers and the matrix also influenced the water absorption behaviour of the composites [16]. Additionally, the water absorption behavior of PLA and PLA composites can be influenced by various factors such as the selection of polymers and fibers, fiber modification methods, water absorption time, and environmental humidity [17].

3.4. Effect of fiber size on tensile strength test

Fig. 9 illustrates the tensile strength of the carbon CCH fiber specimen. The test results indicate a slight decrease in tensile strength for the treated coconut fiber. However, it is worth noting that the specimen exhibited an increase in tensile strength after being mixed with varying sizes of carbon fiber.



Fig. 9. Tensile Strength (MPa) of Pure PLA and PLA/Carbon CCH Composite with Different Fiber Size

As can be seen in Fig. 9, the specimen with pure polylactic acid (PLA) has a higher tensile strength compared to the other 2 samples with the addition of carbon CCH fiber. The carbon fiber sample exhibits fibers of varying diameters, specifically measuring 54 μ m, 75 μ m, and 212 μ m. These fibers contribute

to an enhanced tensile strength, although being smaller in comparison to pure PLA. The inclusion of a filler in the specimen showcases a significant enhancement in flexibility when utilizing pure PLA, but the absence of a filler in pure PLA results in brittleness. According to a scholarly study authored by Zaaba and Ismail, prior research has indicated that pure polylactic acid (PLA) has a greater tensile strength in comparison to composite PLA [18]. According to the findings of Chen et al. (2014) and Neagu et al. (2009), the incorporation of fiber loading has had a significant impact on the microstructure of the foamed cells [19,20].

The observed reduction in the tensile strength of the composite foam can likely be attributed to inadequate interfacial adhesion between the PLA matrix and carbon CCH fiber. This is evident from the SEM micrograph (Fig. 11), which reveals the presence of fiber agglomeration within the PLA matrix, particularly at higher carbon CCH fiber loadings. It is plausible that this agglomeration weakens the bonding between the polymer matrix and fillers. According to Chiulan et al. (2017), the addition of carbon fibers to the PLA matrix can significantly enhance the mechanical properties of the resulting composites [21,22]. It was found that fiber size plays a crucial role in determining the mechanical performance of the composites. In general, smaller fiber sizes tend to improve the tensile strength and modulus of the composites. This can be attributed to the increased interfacial area between the fibers and the matrix, leading to better load transfer and enhanced interfacial bonding. On the other hand, larger fiber sizes may result in a decrease in mechanical properties due to poor interfacial adhesion and stress concentration at fiber ends. Therefore, optimizing the fiber size is critical for achieving the desired mechanical properties in PLA/carbon fiber composites [23].

3.5. Effect of fiber size on flexural strength test

Fig. 10 illustrates the flexural strength of pure polylactic acid (PLA) and PLA/carbon CCH composite, wherein various fiber diameters were employed. In general, it was observed that all specimens experienced failure after the flexural stress reached their maximum value.



Fig. 10. Flexural Strength (MPa) of Pure PLA and PLA/Carbon CCH Composite with Different Fiber Size

Fig. 10 shows that the controlled sample which is pure PLA with no carbon CCH fiber has the higher flexural strength compared to the two samples with the addition of carbon CCH fiber. The flexural strength test of the controlled sample with carbon CCH fiber has decreased due to different fiber sizes. Based on the trend of the graph shows that carbon CCH fiber at a fiber size of 53 µm has the lowest strength, which is 73.59 MPa, compared to other samples. The sample with carbon CCH fiber sizes of 75 µm and 212 µm shows increments to 93.37 MPa and 116.84 MPa, respectively. This is attributed to the fact that larger fiber sizes provide better load transfer and distribution within the material, leading to enhanced mechanical properties. According to a study conducted by Dispenza et al. (2022), researchers varying the fiber sizes had a significant impact on the flexural strength of the composites. They observed that increasing the fiber size resulted in a substantial improvement in the flexural strength of the PLA/CCH composites. The study also demonstrated that incorporating CCH fibers in the PLA matrix further enhanced the flexural strength of the composites. This can be attributed to the interfacial adhesion between the CCH fibers and the PLA matrix, resulting in more efficient stress transfer [24]. Additionally, a study on the physical and mechanical properties of wood plastic composites showed that higher fiber size produces higher strength and elasticity but lower energy to break and elongation [25].

3.6. Morphology analysis

Fig. 11 shows the surface morphology of pure polylactic acid (PLA) at magnifications of $500 \times$ and $2,000 \times$, as depicted in the image. Fig. 11(a) demonstrates the inherent brittleness of the material, as observed in Fig. 11(b), which exhibits minimal elasticity. The observed features, including as the presence of brittle debris, voids, and limited elastic pull-out, are indicative of the usual brittle fracture properties exhibited by PLA.

Fig. 12 illustrates the surface morphology of the PLA/ carbon CCH composite, whereby carbon CCH fibers of vary-



Fig. 11. SEM Images of Pure PLA Samples at Break at (a) 500× and (b) 2.00k× Magnification



Fig. 12. SEM images of a PLA-carbon CCH composite fracture area, demonstrating a debonding of the carbon CCH particle fiber size of a) 53 μ m, b) 75 μ m, c) 212 μ m from the PLA matrix, at 20 μ m with 1000 magnification

ing diameters (53 μ m, 75 μ m, and 212 μ m) were utilised while maintaining consistent filler loading. The composite material containing 15% carbon CCH fiber exhibited a uniform and poorly mixed distribution of the matrix-filler components. The surface of the composite appeared rough, with some filler particles agglomerated within the matrix. This observation suggests that there is a small proportion of carbon CCH filler that does not effectively bond with the PLA matrix. This observation suggests that the filler content in the matrix has exceeded its maximum capacity, indicating that any more filler material would not be able to integrate with the PLA matrix effectively.

Based on the data shown in Fig. 12, it can be observed that the micrograph surface of the scanning electron microscope (SEM) morphology exhibits a consistent 15% fiber loading across all samples. The crack utilised in the tensile testing was crucial in generating the surface morphology. Based on the micrograph analysis, it is evident that the composite's surface exhibits numerous pores, while the fiber structure consists of a limited number of agglomerated fillers, which can be attributed to a higher fiber loading. This phenomenon can be associated with the observed decrease in tensile strength data for fibers with a diameter of 53 μ m.

Fig. 12(c) clearly illustrates the presence of a fractured fiber that is visibly connected to the matrix. However, it is evident that there is a significant abundance of the barrier between fiber and polylactic acid. This is consistent with the water absorption investigation, which revealed that carbon CCH fiber size 212 μ m has a higher water uptake than pure PLA and other fiber size content. It is evident that a significant number of carbon CCH fibers fractured concurrently with the PLA matrix rather than being extracted from the carbon CCH fiber during sample rupture. During the process of maturation, the cells undergo a transformation from an immature state to a hardened and yellowed one. This transformation occurs due to the deposition of a coating of lignin on the cell walls [1].

4. Conclusion

The investigation of the impact of varying fiber diameters in PLA/Carbon Fiber composites on mechanical characteristics was conducted. An investigation was conducted to examine the influence of varying diameters of carbon CCH fibers on physical and mechanical characteristics. Based on this research, it can be inferred that the carbon CCH fiber with a size of 212 mm exhibits the most favourable mechanical properties. The research carried out on the impact of various fiber sizes in PLA/carbon fiber composites has yielded useful insights into the correlation between fiber size and the mechanical properties of these composites. The study has demonstrated that the size of the fibers are crucial in defining the overall performance and robustness of the composites. Enhancing the size of the fibers has been discovered to improve the tensile strength and modulus of the composites by enhancing their ability to transfer loads and adhere at the interface. Conversely, lower fiber diameters have been found to lead to increased fiber breakage, which negatively impacts mechanical characteristics. These findings highlight the significance of adjusting the size of fibers to attain the specific mechanical characteristics in PLA/carbon fiber composites. PLA/CCH composites are known for their biocompatibility and biodegradability. The research on different fiber sizes can contribute to the development of composites that are suitable for biomedical applications, such as tissue engineering and drug delivery systems. In terms of sustainability, the use of CCH as reinforcement in PLA composites contributes to the utilization of a renewable and sustainable resource. Understanding the impact of different fiber sizes on the properties of these composites is essential for promoting their use in various eco-friendly applications. Further research in this area will contribute to the development of advanced materials and the advancement of various industries. The research findings have significant implications for additive manufacturing techniques, particularly in relation to the pivotal role that the mechanical characteristics of composites play. By adjusting the fiber dimensions, the composites can be customized for certain 3D printing applications, hence broadening the potential utilization of PLA/CCH composites in this constantly increasing industry.

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REFERENCES

- S.A.N. Mohamed, E.S. Zainudin, S.M. Sapuan, M.D. Azaman, A.M.T. Arifin, Introduction to natural fiber reinforced vinyl ester and vinyl polymer composites. Nat. Fiber Reinf. Vinyl Ester Vinyl Polym. Compos. 1-25 (2018).
- [2] A. Bengtsson, J. Bengtsson, M. Sedin, E. Sjöholm, Carbon fibers from lignin-cellulose precursors: effect of stabilization conditions. Acs. Sustain Chem. Eng. 7 (9), 8440-8448 (2019).
- [3] K.F. Hasan, P.G. Horváth, M. Bak, T. Alpár, A state-of-the-art review on coir fibre-reinforced biocomposites. Rsc. Advances 11 (18), 10548-10571 (2021).
- [4] S. Farah, D.G. Anderson, R. Langer, Physical and mechanical properties of PLA, and their functions in widespread applications – A comprehensive review. Adv. Drug Deliver Rev. 107, 367-392 (2016).
- [5] R. Siakeng, M. Jawaid, H. Ariffin, S.M. Sapuan, M. Asim, N. Saba, Natural fiber reinforced polylactic acid composites: A review. Polym. Compos. 40 (2), 446-463 (2019).
- [6] I.N. Nasidi, L.H. Ismail, E.M. Samsudin, Effect of Sodium Hydroxide (NaOH) Treatment on Coconut Coir Fibre and its Effectiveness on Enhancing Sound Absorption Properties. Pertanika J. Sci. Technol. 29 (1), (2021).

- [7] A.H. Muhammad, Effect of alkali treatment on the coconut fiber surface. ARPN J. Eng. Appl. Sci. 12 (6), 1870-1875 (2017).
- [8] A. Pérez-Fonseca, H. Teymoorzadeh, J. Robledo-Ortíz, R. González-Nuñez, D. Rodrigue, Polylactic acid composites and composite foams based on natural fibers. Handbook of Composites from Renewable Materials, Structure and Chemistry 1, 25 (2016).
- [9] N.A.A. Hassan, S. Ahmad, R.S. Chen, Density Measurement, Tensile and Morphology Properties of Polylactic Acid Biocomposites Foam Reinforced with Different Kenaf Filler Loading. Sains Malays 49, 2293-2300 (2020).
- [10] G. Rajeshkumar, S.A. Seshadri, G.L. Devnani, M.R. Sanjay, S. Siengchin, J.P. Maran, A.R. Anuf. Environment friendly, renewable and sustainable poly lactic acid (PLA) based natural fiber reinforced composites – A comprehensive review. J. Cleaner Prod. **310**, 127483 (2021).
- [11] A. Rigail-Cedeño, M. Lazo, J. Gaona, J. Delgado, C.V. Tapia-Bastidas, A.L. Rivas, R. Perugachi, Processability and Physical Properties of Compatibilized Recycled HDPE/Rice Husk Biocomposites. J. Manuf. Mater. Process. 6 (4), 67 (2022).
- [12] L.A. Chicos, M.A. Pop, A.M. Zaharia, C. Lancea, G.R. Buican, I.S. Pascariu, V.M. Stamate, Infill density influence on mechanical and thermal properties of short carbon fiber-reinforced polyamide composites manufactured by FFF process. Mater. 15 (10), 3706 (2022).
- [13] E. Kargar, A. Ghasemi-Ghalebahman, Experimental investigation on fatigue life and tensile strength of carbon fiber-reinforced PLA composites based on fused deposition modeling. Sci. Rep. 13 (1), 18194 (2023).
- [14] K.R. Kumar, V. Mohanavel, K. Kiran, Mechanical properties and characterization of polylactic acid/carbon fiber composite fabricated by fused deposition modeling. J. Mater. Eng. Perform. **31** (6), 4877-4886 (2022).
- [15] K. Roy, S.C. Debnath, A. Pongwisuthiruchte, P. Potiyaraj, Recent advances of natural fibers based green rubber composites: Properties, current status, and future perspectives. J. Appl. Polym. Sci. 138 (35), 50866 (2021).

- [16] A. Alawar, A.M. Hamed, K. Al-Kaabi, Characterization of treated date palm tree fiber as composite reinforcement. Composites, Part B. 40 (7), 601-606 (2009).
- [17] Q. Wang, T. Chen, X. Wang, Y. Zheng, J. Zheng, G. Song, S. Liu, Recent Progress on Moisture Absorption Aging of Plant Fiber Reinforced Polymer Composites. Polym., 15 (20), 4121 (2023).
- [18] N.F. Zaaba, H. Ismail, The effect of filler loading on tensile and morphological properties of polylactic acid (PLA)/thermoplastic corn starch (TPCS)/peanut shell powder (PSP) biocomposites. AIP Conf. Proc. 2068 (1) (2019).
- [19] R.S. Chen, S. Ahmad, M.H.A. Ghani, M.N. Salleh, Optimization of high filler loading on tensile properties of recycled HDPE/ PET blends filled with rice husk. AIP Conf. Proc. 1614 (1), 46-51 (2014).
- [20] R.C. Neagu, M. Cuénoud, F. Berthold, P.E. Bourban, E.K. Gamstedt, M. Lindström, J.A.E. Månson, Processing and mechanical properties of novel wood fibre composites foams. Int. Conf. Compos. Mater. (2009).
- [21] I. Chiulan, A.N. Frone, C. Brandabur, D.M. Panaitescu, Recent advances in 3D printing of aliphatic polyesters. Bioeng. 5 (1), 2 (2017).
- [22] N. Van de Werken, H. Tekinalp, P. Khanbolouki, S. Ozcan, A. Williams, M. Tehrani, Additively manufactured carbon fiberreinforced composites: State of the art and perspective. Addit. Manuf. 31, 100962 (2020).
- [23] A. Ramachandran, S. Mavinkere Rangappa, V. Kushvaha, A. Khan, S. Seingchin, H.N. Dhakal, Modification of fibers and matrices in natural fiber reinforced polymer composites: A comprehensive review. Macromol. Rapid Commun. 43 (17), 2100862 (2022).
- [24] C. Dispenza, M.A. Sabatino, G. Infurna, N.T. Dintcheva, Control of end-of-life oxygen-containing groups accumulation in biopolyesters through introduction of crosslinked polysaccharide particles. Polym. Eng. Sci. 62 (2), 426-436 (2022).
- [25] H. Bouafif, A. Koubaa, P. Perré, A. Cloutier, Effects of fiber characteristics on the physical and mechanical properties of wood plastic composites. Composites Part A: Applied Science and Manufacturing 40 (12), 1975-1981 (2009).

732