

Research Article

Experimental Investigations on the Mechanical Characteristics of Natural Fiber Particle-Reinforced Polymer Composites under Cryogenic Environment

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In the past few years, researchers have turned away from previous metals and minerals and more towards lightweight materials throughout addition to creating lightweight, better effective composites for special tasks. The composite materials were created by employing the most advantageous hand layup approach. To attain the aforementioned goals, the following limitations were implemented: at various levels, (i) coconut shell powder, (ii) walnut powder, and (iii) cryogenic treatment hours are all accessible. At 77 K, the laminated composite was put in a cryogenic chamber. Tensile, flexural, and interlaminar shear strengths were all tested mechanically. In comparison to $100 \,\mu$ m coconut shell powder-based particle composites, $80 \,\mu$ m walnut shell composites gave the maximum mechanical strength, while $10 \,$ wt% coconut shell powder and walnut and $80 \,$ wt% polyester also had a beneficial impact. The morphological behaviour of the particulate material was detected using a scanning electron microscope (SEM).

1. Introduction

The growth in ecological sustainability with social response and also new pollutant restrictions or irresponsible oil usage have all led consumers to choose eco-friendly items. Natural fiber is among the most eco-friendly materials in the construction industry, exceeding man-made materials in a variety of aspects. A recent market analysis estimated that the worldwide biocomposite fiber-reinforced industry will be valued at \$2.4 million in 2020 [1]. According to current indications, the global market will continue to rise significantly. Natural fiber-based polymer composites have become increasingly popular in consumer goods and emerging industrial sectors in recent years. Globally, the industry is expected to increase by 12% over the next five years, according to forecasts [2]. Natural fibers are nonsynthetic, non-

man-made fibers. Animals and plants can both provide them. Natural fibers derived from both sustainable and nonrenewable substances, like groundnut, jute, hemp, and cotton, have gotten a lot of consideration in recent decades. Bast fibers (kenaf, hemp, ramie, flax, and jute), as well as all other forms of cellulose fibers, can be classified as "roots and wood" [3]. Composites are flexible, with uses ranging from spacecraft to passenger airliners, fighting planes, and the Space Shuttle. Fiber-reinforced polymer matrices are extremely lightweight, low cost, and less damaging to processing equipment and have relatively high mechanical properties, so they are used in many applications due to the better strength and compensations of organic fibers over man-made fabrics. It is getting a lot of attention. Flexural and tensile strength improved surface polish of molding composites, plentiful recycling, flexibility at certain individual facilities, renewability, and low interface risks [4]. Plant fibers, both tough and gentle, are added to the polymeric materials [5]. Natural filler composites constructed of polymers are becoming increasingly popular because to their inexpensive cost, lightweight, biorenewable nature, and outstanding mechanical properties. Numerous studies have been conducted to take use of the wide range of materials and physical properties of common fillers [6]. The major objectives of such experiments are to alter weight fraction, surface modification, and polymer selection. The scattering condition, interfacial interaction, microstructure, and particulate loading all show a character in the material characteristics of particle-based composites. The dimension of the particles has a pure impact on such mechanical characteristics [7]. At a given nanoparticle concentration, smaller calcium carbonate particles, for instance, offer good strength in filled polypropylene composites. Natural fillers' hydrophilic nature, on the other hand, results in poor interaction with hydrophobic polymer matrices as well as poor dimensional stability when absorption of water stretches fillers [8]. As a result, poor adherence among natural particles and man-made matrices such as polypropylene (PP) is caused by a lack of interaction among fillers and polymer matrices [9, 10]. Furthermore, the heat resistance of organic fillers restricts the use of the composite in hightemperature processing, particularly in extrusion and hot compression procedures. Several organic fillers have been investigated as novel reinforcing elements in polymer-based composite materials, including alfa, coco fiber, muffin, calcium carbonate, mud, and graphite. Walnut shell powder, one of these natural materials, may have a promising future as a novel reinforcing filler in polymer composites. Walnut shells are a flexible, softer abrasion medium with distinct physicochemical characteristics. Such characteristics make it suitable for a wide range of activities, including walnut shell blowing, tumbling, cleaning, filtering, nonskid floors, detergents, and perfumes. Walnut shells are pulverized and processed into conventional mesh sizes ranging from coarse grains to powder form [11, 12]. Heterogeneous composites are made up of many more numerous ferromagnetic layers in a single layer. By eliminating the drawbacks of individual materials, hybridization may enhance the properties of organic fibers and polymeric materials [13, 14]. Coconut plants are frequently produced in the previous period to see



FIGURE 1: Extraction of walnut powder from walnut shell.

the industry's requirement for timber feed. Coconut trees may be found in over 93 countries throughout the world. According to one study, the shipping of coconut oil and other derived goods accounts for more than a quarter of the global coconut sector. As a consequence of the extensive presence of the coconut tree, coconut shells have been one of the biggest pollutants, creating 3.18 million tonnes of toxins annually and contributing to more than 60% of total garbage quantities [15, 16]. The material properties of fiber ceramics could be enhanced by freezing action. Materials used in airframes, for example, must be able to withstand extreme temperatures of up to 210°C. Cooled-down biomaterials and polymers have significantly improved in toughness and stiffness, as well as other properties [17, 18]. As a consequence, cryogenic treatment of compounds could become a significant part of recent investigation and development to enhance the natural fiber behaviour of materials. The primary goal of this research is to assess the effect of combining coconut shell powder and walnut filler on a polyester-based composite material. The organic particulate composite materials are made by hand. The material properties of the fabricated samples were tested following varied lengths of immersion.

2. Experimental Resources and Methods

2.1. Walnut Powder. Gather discoloured walnut shells, rinse them, and then wash them. The walnut shell is cleaned and dried under atmospheric conditions for 60 hours. The walnut shell is next pulverized to reduce its size by roller milling at 400–450 r/min for 8–9 hours. The walnut shell is next finely powdered and sieved to reach particle sizes of 80 and 100 mesh. The extraction of walnut powder from walnut shell is depicted in Figure 1.

2.2. Coconut Shell Powder. Gather discoloured coconut shells, rinse them, and then wash them. The coconut shell is cleaned and dried under atmospheric conditions for 60 hours. The coconut shell is next pulverized to reduce its size by roller milling at 250–350 r/min for 6–7 hours. The coconut shell is next finely powdered and sieved to reach particle sizes of 80 and 100 mesh. The removal of coconut shell powders from coconut shell is depicted in Figure 2.

2.3. Preparations of Particulate Composites. Firstly, an aluminium based mold with a size of $150 \times 150 \times 3$ mm was refined. The matrix material was assorted well with 1 wt% of other curing agents. The hand layup approach was used to construct the composite from walnut and coconut shell



FIGURE 2: Extraction of coconut shell powder from coconut shell.

powder and combinations of both. By hand stirring with a glass rod, varying particle sizes of walnut and coconut shell powder were disseminated in the produced polyester resin. This matrix mixture was scattered over the mold's layers of fibers. The mold was fastened and dried ambient conditions up to 24 h; then, the fiber filaments were completely moistened by matrix mixture. The desiccators were used to keep the fabricated walnut, coconut shell powder, and walnut and coconut shell powder-based materials from captivating any supplementary wetness. Following that, the produced specimens were flooded with liquid N₂ at 77 K for cold healing at various times according to the specification. The treated plates were detached from the cryogenic compartment and maintained at a normal temperature. Table 1 shows the constraints of particulate composites. The weight percentage of matrix is 80 which is common for all the samples. The prepared materials could be utilized in automotives and domestic sectors, and it is shown in Figure 3. Figure 4 demonstrates the photographic view of the hand layup process of composites.

2.4. Testing of Hybrid Composites. For lightweight materials, the produced laminate specimens were cut and conformed to an ASTM specification of D-638-03 mimics for tension, ASTM D-790 for bending, and ASTM D-2344 for ILSS.

3. Result and Discussion

3.1. Tensile Behaviour. Figures 5(a) and 5(b) show the result of tensile behaviour on biocomposites as a role of coconut shell powder, walnut, and hybrids of both powders. The pattern demonstrates a clear improvement in tensile strength for 80 m of particle composites when compared to $100 \,\mu m$ of particle composites. Tensile strengths of 19.84, 21.54, and 25.21 MPa were achieved for $100 \,\mu\text{m}$ of coconut shell powder, walnut, and hybrid composites, correspondingly. The hybrid composites for $80 \,\mu m$ of coconut shell powder, walnut, and bamboo were 21.56, 24.36, and 27.65 MPa, respectively. When individual particles are evaluated, walnut has the highest tensile strength when compared to coconut shell powder. In comparison to 100 m-sized particles, 80 m-sized particles have the best tensile strength. It obviously demonstrates how particle size and strength have an inversely proportionate relationship. When compared to the 100 m size of coconut shell powder and walnut, the 80 m-sized particles were fully dispersed in the polyester matrix in both cases. In comparison to coconut shell powder, walnut shell powder dispersion in the polyester matrix TABLE 1: Constraints of particulate composites.

Sample no.	Weight % and size of walnut and CSP powder
1	20% wt and 80 μ m of walnut powder
2	20% wt and 100 μm of walnut powder
3	20% wt and 80 μ m of CSP powder
4	20% wt and 100 μ m of CSP powder
5	10% wt of walnut, 10% wt of CSP with 80 μm size
6	10% wt of walnut, 10% wt of CSP with 100 μm size

was good. As a result, increasing the interfacial area of coconut shell powder lowered the strength, as did the inadequate interfacial bonding between the particle and matrix polymers. By combining walnut powder with coconut shell powder, the above-mentioned flaws were eliminated. With the loading of both particles, the tensile strength improved marginally. The composites' strength was enhanced as a result of the fiber's ability to withstand pressures transmitted from polymer components [19]. The UTM pictorial of tensile is presented in Figure 5(c).

3.2. Flexural Strength. Flexural loading seems to be the most popular application rate of the deformation technique, which involves stretching a square sample to fracture or using a multiple point flexural evaluation technique. The maximal strain inside the substance at its yielding point is reflected in the flexural properties. The bending strength of particulate biocomposites is demonstrated in Figure 6. When compared to $100 \,\mu m$ particles, $80 \,\mu m$ coconut shell powder and walnut particles have high intensities of 26.95, 31.25, and 33.58 MPa, respectively. Polyester composites' three-point bending strength declined as particle size increased. The addition of coconut shell powder and walnut increased the strength properties of the composite material, with a grain size of $80 \,\mu m$ having the maximum bending strength value. The trends detected in the bending meter of the composite show the high surface area exhibited by the small filler particles, which improves the dispersion in the additives and the matrices and improves the interfacial bond [20]. The SEM image discernibly shows the above trends. Polyester resin has brilliant adhesion to a variety of materials and can be further supported by adding fibers and particles. The strength of polyester is increased with the accumulation of filler, and the best results are achieved with walnut shell filler. The outcomes show that hybrid walnut shells and



FIGURE 3: Application of polymer composites.



FIGURE 4: Hand layup process of walnut and coconut shell powder-based particulate composite.

coconut shell powder-particle composites provide the finest consequences at 10% by weight of filler in both tensile and bending cases compared to other filler fillings.

3.3. ILSS. Interlaminar shear strength (ILSS) is considered a main factor for interlaminar length to depth ratio. Because the failure of a sample is dependent on the above parameters. The interlaminar shear strength of the composite materials increases when the formation of voids decreases. When coconut shell powder and walnut particles are compared, the hybrid (both coconut shell powder and walnut) composites have the highest strength result of 35.78 MPa. Figure 7 demonstrates the interlaminar shear strength behaviour of hybrid composites. It clearly reveals that the filler size and strength have inversional proportional relationships, because increasing filler size reduces the composite strength. This may occur by decreasing the adhesion quality at the interface of the composite materials, particularly at the matrix and its reinforcement phase. Small-sized particles always help to transfer the applied load of the composite materials by means of their strong bonding between the matrices [4].

3.4. Outcome of Cryogenic Handling. In cryogenic processing, the particulate samples were immersed in fluid N_2 at 77 K and went through thermal steering. Under cryogenic

circumstances, Figure 7 illustrates the tensile, flexural, and interlaminar shear strength strengths of coconut shell powder and walnut powder-based hybrid composites. At 30 minutes of cryogenic treatment for $80\,\mu m$ particle size, the greatest value of tensile strength is 31.69 MPa, 35.61 MPa of flexural, and 35.87 MPa of interlaminar shear strength, as revealed in Figures 8(a) and 8(b). Similarly, 100 μ m- sized particles with 30 minutes of cryogenic treatment had the greatest tensile, flexural, and ILSS values of 29.03 MPa, 33.02 MPa, and 34.30 MPa, respectively. As a result, the $80 \,\mu m$ particles were equally scattered in the polyester matrix as a result of this. In all cases (80 and 100 μ m), following 30 minutes of cold working, the plurality of a composites was clearly noticeable, showing its superior mechanical capabilities. It could be owing to latent stresses caused by compressive connection like a consequence of freezing stressing as in nanocomposites. Latent strains arise as colder concentrations because of variable matrix shrinking. These compressed interfacial strains aid in particle-matrix interaction and adherence, resulting in greater outcomes.

At low temperatures, cryogenic healing of manufactured composites makes the elements stronger. As the rigidity of the specimen reduces, so does its elasticity, resulting in a reduction in deflection. The delamination resistance of coconut shell powder and walnut hybrid composites was



FIGURE 5: Tension behaviour of (a) 80 µm; (b) 100 µm particles of coconut shell powder, walnut, and hybrid (c) UTM for tensile testing.

improved after cryogenic treatments owing to the increase in compression pressures at the boundary. The mechanical characteristics of composite materials degrade after 30 minutes of exposure. Due to the higher amount of fiberresin imbalance, lengthier fluid nitrogen conditions whitethorn result in greater heat conductance. Because of the decreased contact, decomposition characteristics are additionally damaging to the coconut shell powder and walnut systems. It has a significant decrease in the duration to remedy matrix. Large interface debonding zones are common in low-bonded composites, which raises a slew of menace influences that can lead to the propagation of crack [1, 5].

3.5. Fractographic Study. The SEM equipment was used to evaluate the fractographic characteristics of coconut shell

powder and walnut-based hybrid composites. Figures 9(s) and 9(b) show the 80 μ m and 100 μ m sizes of coconut shell powder and walnut-based hybrid composites in a liquid nitrogen environment for varied periods of time. It has been discovered that reducing the filler size increases mechanical strength. This may be related to the effect of increasing the bonding between the filler and the matrix material at the interface. The shipment of applied load capacity between the matrix and the filler material and the good interfacial bond between the filler and the matrix were factors that led to improved composite strength. The increased value of interaction at the bounty surface was displayed by the 80 μ m particle size, which resulted in enhanced dispersion quality of filler in the matrix and shown in Figure 9(c) [10, 12, 16].



FIGURE 6: Flexural strength of (a) 80 μ m and (b) 100 μ m particles of CSP, walnut, and hybrid.



FIGURE 7: Interlaminar shear strength of (a) $80 \,\mu\text{m}$ and (b) $100 \,\mu\text{m}$ particles of CSP, walnut, and hybrid.

Because of the increased compressive shrinkage stresses at the border, the delamination resistance of coconut shell powder and walnut hybrid composites is enhanced following cryogenic treatments. After 30 minutes of exposure, the mechanical properties of composite materials worsen. Lengthier cold processing durations may result in more heat conductance because of the higher amount of fibermatrix disparity. Decomposition structure are very harmful to the coconut shell powder and walnut composite systems due to the reduced contact. This is clearly demonstrated in Figure 9(c). According to SEM, the homogeneity between the coconut shell powder and walnut particles and the matrix



FIGURE 8: Cryogenic treatment effect of mechanical strength (a) $80 \,\mu\text{m}$ and (b) $100 \,\mu\text{m}$.



FIGURE 9: SEM images of (a) $80 \,\mu\text{m}$ (b), $100 \,\mu\text{m}$, and (c) $80 \,\mu\text{m}$ particle in 45 min cryogenic treatment.

declines as the particle size increases. This explains why the strength of the composite decreases as the particle sizes inside the matrix structure get larger.

4. Conclusion

The following are the results of this experimental investigation activity:

(i) In comparison to $80 \,\mu\text{m}$ of coconut shell powder particle, $80 \,\mu\text{m}$ of walnut provides a maximum mechanical strength of 21.56 MPa in tensile, 26.95 MPa in flexural, and 28.96 MPa in interlaminar shear strength, whereas for $100 \,\mu\text{m}$ of coconut shell powder particle, $100 \,\mu\text{m}$ of walnut provides the maximum mechanical strength of 21.54 MPa in tensile, 30.25 MPa in flexural, and 27.96 MPa in inter laminar shear strength. Compared to individual particle-based composites, hybrid composites provide the highest mechanical strength, with 27.65 MPa of tensile, 29.68 MPa of flexural, and 35.78 MPa of interlaminar shear strength. Compared to large-sized particles, small-sized particles are evenly distributed throughout the polyester matrix. It helps to reduce the formation of voids

- (ii) The cryogenic treatment provides a high amount of residual stress in the composite interface, which helps to increase the interfacial bonding between the reinforcement and the matrix. However, it was only effective for 30 minutes of treatment. After that, strength was reduced due to high stress on the composite surface
- (iii) In future work, other experimental testing like bending, compression, and shear test may be included for further inclusion in general applications

Data Availability

The data used to support the findings of this study are included within the article. Further data or information is available from the corresponding author upon request.

Conflicts of Interest

The authors state that the publishing of this paper does not include any conflicts of interest.

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