Research Article

Interlaminar Shear, Bending, and Water Retention Behavior of Nano-SiO₂ Filler-Incorporated Dharbai/Glass Fiber-Based Hybrid Composites under Cryogenic Environment

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Received 27 May 2022; Accepted 19 July 2022; Published 30 July 2022

Academic Editor: Lakshmipathy R

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In current history, adding nanoscale and micron-sized filler materials to composite materials for fabrication has been a popular approach for improving the composite’s mechanical characteristics. Due to their lower friction coefficient, excellent mechanical strength modulus, and low moisture uptake, filler-based hybrid composite materials are replacing metallic materials. Glass/Dharbai hybrid composites with nano-SiO₂ fillers have been created in this study. After manufacture, the composite materials were treated with liquid nitrogen at 177 K for various durations. Every sample material was cut according to ASTM standards to investigate mechanical features such as ILSS, impact test, and flexural strength. The broken composite specimen was studied using a scanning electron microscope. Water retention studies have been conducted under two distinct liquid solutions: tap or regular water and seawater. ILSS, flexural strength, and water retention were all greater in 4 wt.% of nanofiller-rich composites than in ordinary composites. Compared to 30 minutes, the 15-minute cryo-treated specimens provide the highest mechanical strength. On the other hand, the automobile, aviation, and shipbuilding sectors would benefit from a nanofiller-based composite.

1. Introduction

Several academics are interested in using these hybridized composite materials because of their lighter weight, low price, easily available, low density, and high strength modulus, making them a popular structural material in recent times. The natural fiber is a low-cost substance readily accessible in the environment and is a renewable material [1]. Natural materials outperformed synthetic fibers in terms of accessibility, sufficient elastic strength, lightweight, cheap, reduced energy need, biocompatibility, fewer environmental risks, recycled materials, and environmental friendliness [2]. Natural fibers’ shortcomings, like poor dimensional accuracy and robustness, increased hygroscopic, poor hydrophilicity, rapid aging at moderately raised temperatures, and inability with hydrophobic adhesives, limit their use in high-end products [3]. Creating a pollution-free and ecologically sound civilization has emerged from the contemporary world and its shifting requirements [4]. It can be accomplished partially or completely by changing the materials
used to construct structures, enterprises, and substances used in various applications [5]. Conventional metals are the primary carbon dioxide emissions when processed or deteriorated.

Furthermore, the globe today needs ecologically friendly biomaterials. As a result, scientists are focusing their efforts on creating a new biodegradable natural chemical. In this instance, biomaterials are one of the finest options for overcoming the metal and alloy shortage [6]. Biocomposite components are light, have distinct mechanical characteristics, and are cost-effective with environmentally friendly. Ceramics and alloy steels cannot give the unique combination of material qualities required for recent breakthroughs, particularly in aircraft, submarines, and transportation [7]. Materials used in aviation frequently fail to fulfill the high fracture toughness and relative stiffness required by engineering constructions. Composite material assists in reaching the desired quality. Because they are often robust and stiff, these materials are suitable for various technological applications [8]. Polymer composite materials have been the most popular new materials in recent history, with uses in project management and other structural features. In terms of material preparation, natural fibers have various advantages over synthetic materials, including being recyclable, having the lowest density, being less costly, having greater flexibility, and providing longer durability [9]. Natural fibers are very inexpensive and have excellent mechanical qualities. Natural fibers are used to make a variety of fabrics, cables, canvasses, and sheets. Natural fiber sources have been detected in plant components such as stems, leaves, seeds, and fruit. The digging and tensile characteristics of vakka and date fiber-reinforced composites were examined, which led to the possibility of using these fiber-reinforced composites in lightweight products.

On the other hand, natural fibers are the essential component of fiber-reinforced composites, as they bear most of the load [10, 11]. As a result, the mechanical characteristics of the composite are influenced by the suitable choice of fiber reinforcement, fiber length, and fiber weight concentration. Dharbai (Eragrostis cynosuroides) is a popular plant in India’s southern and northern areas. No attempt has yet been made to determine the fiber content of this shrub. As a result, this research is aimed at seeing if Dharbai fibers may be employed as a reinforcement in epoxy matrix composites [12]. Natural fibers have different potential due to differences in cellulose, hemicellulose, and lignin chemical components. The fiber and matrix must mix and have inclinations in order for the composite to have superior qualities [13].

Extremely thin glass strands are used to make fiberglass. To make the fiberglass, tiny silicon threads and various glass compositions were pressed together under tremendous pressure [14]. Carbon, as well as other polymer fibers, is more expensive and fragile. It can be used to fabricate composites to enhance their strength while reducing their load. The most common glass used in the manufacture of matrix materials is glass fiber-reinforced plastics, often known as ‘glass fiber’ and “E-glass.” Tension and compression stress are not a problem for fiberglass. High-strength textiles, highly corrosive textiles, insulation, soundproofing, and electromagnetic shielding are all made with fiberglass [15]. Fiber-reinforced plastic containers and containers are commonly made with these materials. Synthetic fibers are inserted into the matrices to counteract the deficit of natural fibers, resulting in the manufacturing of hybrid nanocomposites [16]. It was discovered that after creating a hybrid composite matrix, the manufactured composites have the integrated qualities of their subsequent hybrid matrix. The advantages of a mixed matrix over its counterparts are that the disadvantages of one fiber may be accounted for by the advantages of other fibers [17]. The configuration of each fiber, the direction of the fiber, the thickness of each fiber, the aspect ratio of fiber loading, characteristics of fibers, breakdown stress of fibers, and the level of interaction with the system of fibers all impact the mechanical characteristics of hybrid composite materials [18]. On natural fabrics, the chemical treatment would’ve had two significant consequences. (i) It increases the proportion of cellulose visible on the fiber’s exterior and (ii) raising the number of possible reagents. It improves the outer quality of the fibers, enhancing structural adherence among all fibers and matrix. Processed coir fibers in solutions at different NaOH levels (5% and 10%) and discovered significant solubility whenever the fibers were exposed to a 5% sodium hydroxide solution. According to Rana et al.’s [19] analysis of the effects of NaOH accumulating (1, 3, 5, and 7%) on a material typical of sisal-based composites, the 5% alkali-treated composites have the maximum tensile strength. When kenaf fiber composites were processed with NaOH (5, 10, and 15%), Nam et al. [20] discovered that the tensile strength, flexural strength, and impact strength all increased by 13%, 14%, and 30%, respectively, at the lowest NaOH concentration. Unfavorable results happen as the level of NaOH increases. The high levels cause the fibers to crystallize, which makes them rigid and brittle and causes high resistance and constrained expandability. These textiles’ enhanced brittleness caused them to shrink more under stress, which decreased the material’s properties and prevented them from transmitting stress at the boundaries effectively. As a result, in the ongoing investigation, 5% NaOH levels were used.

Hybridization is an innovative process that may be used to manufacture composites to produce enhanced mechanical and other matrix characteristics. The use of fillers in hybrid composites increases the composite’s properties. Improved mechanical characteristics and surface quality of manufactured composites are best achieved using nano- or micro-sized spherical fillers [21, 22]. The use of nano-
and microalumina filler materials in the ship, aircraft, and automotive sectors are growing, as these composites have a low wear rate and great strength. Wear is a major concern for pistons, connecting rods, piston heads, and structural frames in automobiles and aeroplanes, so nanoscale and microscopic composite matrices are frequently used for these sectors [23]. According to Barkoula et al. [24], sizing agents substantially impact carbon fiber matrix because they have a higher molecular weight and operate as a connection between carbon fiber and matrix resin. Because nano- and microfillers absorb less water, they can be employed in the shipbuilding industry. Epoxy resin is a reinforcing material in composite fabrication because it provides greater strength, flexibility, chemical stability, and superior electrical performance. The laminated grade impacted the empty content. Because the lamenting quality is low, it increases the void content, which is attributable to the composite’s poor mechanical qualities [25]. Cryogenic behaviors can increase the mechanical characteristics of fiber-reinforced composites. For instance, resources used in aeroplane construction should withstand severe temperatures of up to 200°C. Composites and plastics that have been cryogenically treated show high strength, tougher, and consume improved rigidity and wear confrontation [26]. As a result, liquid nitrogen treating of composites can develop an important aspect of ongoing investigation and growth to enhance natural composite material behavior.

According to the reviewed literature, relatively no studies on nano-/microfiller-based nanocomposite under cryogenic settings exist. Furthermore, there is no comparison investigation of mechanical and water retention capabilities of nanoscale filler composites accessible today. In light of these gaps in the literature, the current study focused on comparing the mechanical and water-absorbing capabilities of nanoscale silica filter-based glass-Dharbai composites. This research will undoubtedly benefit the maritime, aviation, automobile, and windmill manufacturing businesses and researchers. The novelty of the current work is highlighted by the fact that this would be one of the first investigations to evaluate nanocomposite employing weaved Dharbai as reinforcements and nanoscale silicon as fillers in a polymer matrix under cryogenic conditions. The results of this study are anticipated to expand the use of natural fiber-based composites at subzero temperatures. The overall combined impacts of the composite’s cold working and piling order on the mechanical properties of Dharbai/glass fibers were studied for the first time.

### 2. Experimental Works

#### 2.1. Materials

Rithu Fiber Industry in Coimbatore, Tamil Nadu, India, provided the Dharbai and glass fiber reinforcement materials. The reinforcing materials and matrix system are shown in Figure 1. Nanosilicon oxide particles and an epoxy matrix were employed in this study. Naga Chemicals Industries provided the matrix and nanofillers in Chennai, Tamil Nadu, India. The fibers were thoroughly laved with clean water to remove the moisture and sun-dried for two days. A knife is used to trim the Dharbai leaves, which are then allowed to dry for 3 days in the shade. Following an 8-day water retting period, the fibers were cleaned to eliminate any accumulated foreign contaminants. The remaining moisture in the fibers was subsequently removed by drying them at room temperature for two days. The Dharbai fiber mat was subsequently created using these dried lengths of fiber.

#### 2.2. Alkaline Processing

The fiber from the Dharbai stem was removed using the water retting process. Both fibers were treated with a 5% NaOH solution in the trays. The fibers were soaked in the mixture for 4 hours for optimal results. After extracting the soaked fibers from the mixture, they were carefully rinsed to remove any leftover alkaline solution. A final wash with distilled water was used to

### Table 1: Nanocomposite preparation and their parameter levels.

| Symbol | Combinations                        | Nanoparticle additions (wt.%) | Cryogenic treatment (min) |
|--------|-------------------------------------|------------------------------|--------------------------|
| A      | Dharbai-glass-Dharbai & glass-Dharbai-glass | 0                            | 15 and 30 min            |
| B      | Dharbai-glass-Dharbai & glass-Dharbai-glass | 2                            | 15 and 30 min            |
| C      | Dharbai-glass-Dharbai & glass-Dharbai-glass | 4                            | 15 and 30 min            |
| D      | Dharbai-glass-Dharbai & glass-Dharbai-glass | 6                            | 15 and 30 min            |

![Flexural strength values of Dharbai/glass/nanosilica-based hybrid composites.](image)
2.3. Preparation of Nanocomposites. In the first step, the nanosilica and epoxy were blended using a mechanical churning technique for 15 minutes to combine the matrices and fillers. Using ultrasonic vibrations, the ultrasonicator is then used to spread the filler into the matrix. Several weight proportions of nanosilica filler loading were employed to form a nanocomposite, including 2, 4, and 6 wt. %. The nanosized silica and epoxy mixture was put in a glass pipette, stirred physically, and held for 45 minutes in an elevated ultrasonic bath on pulse mode. The nanocomposites were made in a steel mold measuring 150 × 150 × 3 mm. Wax was initially put into a mold to make it simpler to separate the composite lamination. Matrices comprise a 10:1 ratio of resin to a curing agent [29]. Fillers varying in weight from 1.5 to 4.5 grams and of equal dimension were added to the mixture (micro). To ensure full mixing, the solution was spun continuously for 10 minutes. In this case, 40% of the solution was put into the mold first and then treated (oven-dried) fibers, and lastly, the remaining matrices were poured over the fibers. The grid was evenly applied to all four edges with a roller. 12 kg of stress was sustained on the mold to maintain lamination width and eliminate extra matrices, resulting in a 3 mm composite with a restricted size. The mold was placed in a 75°C microwave oven for 3 hours to dry the laminate adequately. The lamination was then divided into pieces and tested according to ASTM standards. Table 1 shows the nanocomposites’ parameters and their levels.

2.4. Cryogenic Treatments. A programmed, heat-regulated cryogenic container was used for the cryogenic procedure. A regulated cooling pace was used to reduce the temperature to 196°C. According to the design of the experiments, the manufactured specimens were subsequently submerged in liquid N₂ at 77 K for cryogenic treatment for varied periods (15 and 30 minutes). The composite materials were warmed back to room temperature using a regulated, consistent rate of heat after processing.

2.5. Characterizations. The biobased nanocomposites were cut to ASTM D-790 dimensions (width 10 mm, length 125 mm, and thickness 3 mm) for flexural testing and ASTM D 2344 replicas with dimensions of 45 × 3 × 3 mm for interlaminar shear strength. At a microscopic level, SEM was employed to analyze damaged composite materials. To increase the electrical conductivity of the composites, the specimens were laved, dried, and surface coated with 10 nm gold before SEM clarity.

2.6. Water Retention Behavior. Composite materials were blended according to ASTM D570 to form rectangular samples measuring 39 mm × 10 mm × 3 mm. The water retention test uses three fluid media: tap water and seawater. For 60 days, all two specimens were collected in each fluid.
Before putting the specimens in fluid, their initial weight is determined. The weight of each specimen is assessed every fifteen days to evaluate weight changes caused by hygroscopic. The period between removing samples from fluid and weighing them is quite brief. Adhered fluid on composites is cleaned off with new cotton before a measure of weight. The hygroscopic percentages were calculated using the formula below [30].

\[
\text{Moisture absorption} = \frac{W_2 - W_1}{W_1} \times 100
\]

where \( W_2 \) is the sample’s weight after immersion and \( W_1 \) is the sample’s weight before immersion. Five tests were conducted on each sample type, with the average findings presented.

### 3. Result and Discussion

#### 3.1. Bending Characteristics of Nanocomposites

Figure 2 shows the bending strength of nanocomposites. Internal stress generated in a substance is characterized by its flexural. Flexural strength is the tension exerted right before breaking [31]. It is also known as the rupture modulus. The lower the deflection, the higher the flexural strength. The mechanical characteristics of the uttermost fiber layer of the composite matrix are used to compute the modulus of rupture [32]. The composite material was subjected to compressive and tensile stress simultaneously, and most samples failed to owe to tensile stress [33]. The failure starts on the side of the beam under stress and slowly spreads upward [34]. Figure 2 shows the results of the flexural strength tests conducted. The flexural modulus of 4 wt (C type). Percent nanosilica is the highest, compared to 2 and 6 wt. % and unfilled composites (A, B, and D types), respectively [35]. Compared to various weight percentages of silica, nanocomposite has a 10.23% higher flexural strength and a 25.69% higher flexural strength than normal-composite [36]. When compared to other weight percentages of silica and normal composites, the 4-weight percentage of silica added nanoparticle enriched filler is best for fabricating the composite because it increases the interfacial bond strength and minimizes the likelihood of voids and fiber pull out.
microcracks forming [37]. As a result, the flexural strength of nanocomposites is pretty high. As the quantity of silica particles in a microcomposite increase, microscopic gaps emerge in the interfacial region, which aid in the creation of microcracks [38].

As a consequence, the flexural strength of composite decreases when compared to 4 wt.% nanocomposite. According to the research, the outer layer comprises Dharbai fiber, which is weaker than glass [39]. The existence of larger amounts of lignin and hemicellulose in Dharbai fiber, which does not form a strong link with the matrix, is the source of the finding mentioned above shown in Figures 3(a) and 3(b) [40]. As a result, it is unable to withstand stress and readily breaks when subjected to it. The composite’s toughness has decreased due to the filaments splitting and delamination [41].

3.2. Interlaminar Shear Strength. The ILSS response of composites is used to assess if the material shears between its layers. The ILSS test analyzes layer bonding in composites to determine whether they can tolerate shear pressure at a specific spot [42]. Compared to the first and third levels, the C levels (such as 4 wt. % silicon) produce the highest ILSS values (such as 2 and 6 wt. % silicon). Increasing the quantity of nanosilicon in composites enhances interlaminar shear strength, which is self-evident [43]. However, ILSS was extremely rich in silicon at 4 wt%. Figure 4 demonstrates the ILSS values of nanocomposites. Increased silicon concentration in the matrix enhances matrix bonding, which leads to improved strength qualities [44]. The functional groups on the nanosilicon–oxide interface enhance the quantity of cross-linking in the samples, which improves the shear behavior [45]. The initial results, however, are only applicable up to a weight of 4%. The mechanical strength reduces when the silicon powder concentration grows over certain limits [46]. Poor silica particle dispersion in the epoxy matrix might be to blame. The composite outer layer followed the same pattern as the flexural outer layer [47]. SEM indicates it. The exterior glass fiber hybrid nanocomposites exhibit a lower amount of fiber pulls out than the Dharbai fiber exterior shown in Figure 5.

It has determined whether there are shear behaviors between the layers of the materials, and mixtures are tested using the ILSS reaction [48]. It has determined the degree of binding among layers necessary to withstand shearing stress at a specific location; the composite is subjected to ILSS testing [49]. Delamination among layers significantly impacts the breaking modulus and bend displacement among plies. When the single layer is under load, the failure of composite layer results in a tragic disaster. The ILSS examination is used to evaluate this failure. The stronger the ILSS value, the more strongly the nanofiller, matrix material, and Dharbai fibers adhere to one another [50].

3.3. Effect of Cryogenic Treatment. During cryogenic treatment, the composite plate was exposed to liquid N$_2$ at -196°C and thermal stress. Cryogenic treatment of polymer-based composites is an innovative way to improve mechanical properties. Figures 6 and 7 show the effect of cryogenic handling on the mechanical properties of polymer-based hybrid composites. The extreme flexural properties of 7372.16 MPa and the ILSS of 6359.26 MPa were achieved after 15 minutes of treatment, as shown in Figures 6 and 7. It might be due to residual stress induced by the compression contact due to the composite materials’ cryogenic straining. At low temperatures, residual stresses are formed due to changing matrix and fiber shrinkage. The interface tensions above help maintain fiber and matrix contact and improve adhesion, resulting in superior results [26, 31, 32]. The microstructural image of nanocomposites after cryogenic treatment at 15 min and 30 min is showed in Figure 8. The order in which fibers are stacked significantly impacts mechanical characteristics. Glass fiber exterior-based nanocomposites have the best mechanical strength in cryogenic temperatures compared to Dharbai fiber exterior. Glass fiber composites were used to control the formation of residual tension at the exterior. It cannot, however, be used on Dharbai fiber exterior composites.

Figure 8: Microstructural image of nanocomposites after cryogenic treatment (a) 15 min and (b) 30 min.
because the number of legionellosis elements in Dharbai fiber is higher [51].

3.4. Hygroscopic Characteristics. Natural fibers can accumulate humidity when they come into contact with it. The inclusion of hemicellulose in the fiber promotes the retention of water. They are moisture-resistant, attributed to the prevalence of hydrophilic hydroxyl groups and lignin. Swelling occurs as the fibers accumulate water until the fiber units are filled. Water destroys the bonding resin, reducing the adhesion in the matrix and resulting in lower flexural and ILSS properties. Higher moisture content causes the matrices to enlarge/bulge, plasticize, and fracture, which increases the passage of water phenomena into the matrix and degrades mechanical and thermal characteristics. The moisture of composite materials under distinct stacking sequences is investigated in two different fluids in the current study (tap water and river water). Figure 9 shows the relationship between time duration (days) and water retention (%) for tap water and Vaigai river water, respectively. Compared to the Dharbai fiber outside, the glass fiber exterior with a 4 wt. % nanomatrix contributed the least water content [23, 33]. It occurs because nanosilica has a larger surface area than Dharbai composites, resulting in better

Figure 9: Water retention behavior. (a, b) Dharbai fiber exterior specimen. (c, d) Glass fiber exterior specimen.
interface bonding between the matrix and fiber. Dharbai composites include bigger silica particles, which can cause gaps, allowing water to easily permeate into the interface bond via capillary action, resulting in a greater water retention rate than glass exterior/nanocomposites [34–36].

4. Conclusion

The mechanical properties of Dharbai/glass/nanosilica-reinforced epoxy-based hybrid nanocomposites were investigated using the traditional hand lay-up method according to ASTM standards. Some observations are as follows:

(i) Mechanical properties of nanosilica inclusion are greatly improved due to the ultrasonic swirling process used in composite manufacture, which assists in properly spreading silica fillers in hybrid composites.

(ii) The mechanical properties of hybrid composites are decreased after 4 wt.% filler addition due to inappropriate soaking of the fillers and problems in fiber/filler interaction. The composites with the highest flexural and ILSS were type C specimens with 4 wt.% silica, 15 minutes of cryogenic treatment, and glass fiber exterior-based composites.

(iii) SEM tests show that the ultrasonic stirring approach resulted in proper fiber dispersion in nanocomposites. According to the findings, nanosilica particles have good adhesive strength and chemical compatibility with epoxy, Dharbai, and glass-based hybrid composites.

(iv) In terms of hygroscopic, C type model composites with a 4 wt.% addition of nanosilica outperform A, B, and D type model composites. When compared to river water, the composites had better hygroscopic capabilities.

(v) Compared to the Dharbai exterior specimen, the glass fiber exterior specimens reveal the highest mechanical and water retention characteristics.

The study covered the in-depth experimental effort to determine the characteristics of Dharbai/nano-SiO₂. However, it is acknowledged that the creation of natural composites in a cold environment involves several difficulties and affects the caliber and characteristics of the materials. For manufacturers, choosing a reliable production method to create composite materials for cryogenic applications is still challenging.

Data Availability

The data used to support the findings of this study are included in the article. Should further data or information be required, these are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

The authors thank the Saveetha School of Engineering, SIMATS, Chennai, for the technical support to complete this research work. The authors appreciate the supports from Ambo University, Ethiopia.

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