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Retraction

Retracted: Statistical Analysis on Interlaminar Shear Strength of Nanosilica Addition with Woven Dharbai/Epoxy Hybrid Nanocomposites under Cryogenic Environment by Taguchi Technique

Adsorption Science and Technology

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This article has been retracted by Hindawi following an investigation undertaken by the publisher [1]. This investigation has uncovered evidence of one or more of the following indicators of systematic manipulation of the publication process:

(1) Discrepancies in scope

(2) Discrepancies in the description of the research reported

(3) Discrepancies between the availability of data and the research described

(4) Inappropriate citations

(5) Incoherent, meaningless and/or irrelevant content included in the article

(6) Peer-review manipulation

The presence of these indicators undermines our confidence in the integrity of the article's content and we cannot, therefore, vouch for its reliability. Please note that this notice is intended solely to alert readers that the content of this article is unreliable. We have not investigated whether authors were aware of or involved in the systematic manipulation of the publication process.

Wiley and Hindawi regrets that the usual quality checks did not identify these issues before publication and have since put additional measures in place to safeguard research integrity.

We wish to credit our own Research Integrity and Research Publishing teams and anonymous and named external researchers and research integrity experts for contributing to this investigation.

The corresponding author, as the representative of all authors, has been given the opportunity to register their agreement or disagreement to this retraction. We have kept a record of any response received.

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[1] M. Ponnusamy, L. Natrayan, P. P. Patil, G. Velmurugan, and Y. T. Keno, "Statistical Analysis on Interlaminar Shear Strength of Nanosilica Addition with Woven Dharbai/Epoxy Hybrid Nanocomposites under Cryogenic Environment by Taguchi Technique," *Adsorption Science & Technology*, vol. 2022, Article ID 6571515, 9 pages, 2022.

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Research Article

Statistical Analysis on Interlaminar Shear Strength of Nanosilica Addition with Woven Dharbai/Epoxy Hybrid Nanocomposites under Cryogenic Environment by Taguchi Technique

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Biocomposites are becoming more popular due to their capacity to replace artificial materials at a lower cost while enhancing environmental responsibility. In contrast, biocomposites have poor mechanical and interface properties. This research is aimed at determining the interlaminar shear strength of composite materials reinforced with Dharbai fibre and nanosilicon powder. The composites were made using a hand lay-up method with the following conditions: (i) weight % of nanosilica filler, (ii) thickness of fibre mat, and (iii) cryogenic treatment period, each at three different levels, to meet the goals mentioned above. The composites were laminated using a traditional hand lay-up method, and their interlaminar shear strength was determined using the ASTM standard. According to a recent study, nanocomposites containing 4% nanoscale silicon and 300 grammes per square metre of woven Dharbai fibre showed the highest interlaminar shear strength after 15 minutes of cryogenic treatment. Fibre content increased the mechanical properties of pure epoxy in general. As the fibre and filler concentrations grew, more energy was required to break the fibre bundles between the matrix and its resin. According to the ANOVA, the cryogenic treatment was the most significant factor, contributing up to 59.58%, followed by woven Dharbai mate, contributing 22.11%, and nanosilicon at 18.30%. SEM is used to investigate the cracked composites' fractographic examination.

1. Introduction

Composite materials are getting popular and interested in developing materials and technology due to their better structural features and developing environmental issues such as climate change and contaminants. The physicochemical characteristics of the fibre vary as the concentration of constituents such as lignocellulosic biomass, pectin, lignin, and wax varies [1, 2]. Natural composite material is appropriate for various applications such as wrapping, construction, and automobile dashboards, due to inherent traits such as lighter density, cheap cost, great mechanical capabilities, and sufficient flexibility. Natural textiles, unlike synthetic materials, are recyclable, have no negative consequences, are easier to work with, and bear less weight [3, 4]. Furthermore, to improve the strength of mechanical features of composite materials, cellulose fibres could be combined with manufactured fibres such as glass and carbon [5]. Dharbai (Eragrostis cynosuroides) is a commonly planted plant in both the southern and northern areas of India, and it produces a range of natural fibres. No attempt has yet been made to analyze this shrub for its fibre. As a result, this study is aimed at determining if Dharbai fibres can be used as a reinforcing material in epoxy matrix composites [6]. Due to variances in cellulose, hemicellulose, and lignin chemical components, natural fibres have varying potential. The fibre and matrix must interact and have affinities for the composite to have better properties.

Dharbai fibres are lignocellulosic fibres containing OH compounds that absorb water quickly and decrease the performance of natural composites, especially in terms of dimension stability [7]. Fresh untreated filaments must be pretreated with artificial chemicals to maximize effectiveness. It has eliminated extra cellulose, pectin, waxes, and hydrocarbons. It has critical because lignin, pectin, and lignocellulose function similarly, resulting in the bonding of microfibrils and the joining of neighbouring fibres to create a grouping. All this interferes with the proper dispersion of fibres within the polymer [8]. Sodium hydroxide (NaOH) is one such alkaline that is widely employed since it leaves no pectin remnants in the fibres following the alkali treatment. This also destroys the ethereal bonds by acidic breakage and eliminates them from the fibre surface. According to previous research, if unprocessed filaments are processed using 5% sodium hydroxide, the composites' load-carrying ability increases severalfold compared to composites generated from untreated fibres [9, 10]. The chemical treatment causes surface quality, facilitating binding between the fabrics and matrices. This can reduce the filamentous quantity by the oxidizing level, causing the crystalline phase of the finished good to be disrupted. It is advised that, following alkalization, the fibres be washed with purified water to remove any surplus caustic. After that, acidity processing with caustic soda is advised since investigations have shown that neutrality fibres possess excellent mechanical properties [11, 12].

One of the most trustworthy and recognized composites for producing models in the modern environment of materials and industrial engineering would be those built using nanoparticle-reinforced polymers [13]. Several studies have demonstrated that when nanocrystals are employed for reinforcing, the synthetic behavioural qualities increase more than the mechanical characteristics. Because of its remarkable electromagnetic, structural, and thermodynamic capabilities, nano-silicon oxide is another imperative element used for reinforcements [14, 15]. It has a solitary organism's width and a two-dimensional hexagonal design. It is widely employed in electrical and optical gadgets [16, 17]. Due to its durability, it has become an excellent filler in epoxy restorative materials. The presence of an oxide position in nanosilica material that functions as a powerful adhesive throughout the matrices is the primary explanation. The technique of blending nano-silicon oxide nanopellets with the polymer matrices is known as ultrasonic irradiation [18]. Cryogenic properties can improve the mechanical properties of fibre-reinforced composites. For example, materials used in aircraft construction should tolerate extreme temperatures of up to 200°C.

Cryogenically treated composites and polymers have great strength, are more robust, and have increased stiffness and wear resistance. As a result, liquid nitrogen treatment of composites may become an essential part of ongoing research and development to improve natural composite material properties [19]. Taguchi parameter design helps streamline production processes and achieve high-quality goals while keeping costs low. The grey-based Taguchi approach was used to refine the parameters of several multi-response problems in the manufacturing system. Throughout this method, an orthogonal design of array (OA) is developed to assess a number of variables in reduced trials, and the S/N ratio is employed to assess the influence of noise components on target attributes. The S/N ratio consolidates the various data points inside an experimental, according to the research feature [20]. Three sorts of performance need to be considered when examining the S/N ratio: short the better, larger the better, and nominal the better.

The novel aspect of the current study can be highlighted by the fact that this is one of the first studies to investigate composites with woven Dharbai as reinforcement and nanosilica as filler material in epoxy resin under cryogenic conditions. It is envisaged that the results of this investigation could open up new application routes for natural fibre-based composites in extremely cold environments. The primary purpose of this research is to construct, test, and improve the mechanical properties of natural hybrid composites utilizing the following criteria: weight % of nano-silicon, the thickness of Dharbai fibre in grammes per square metre, and cryogenic treatment duration. Natural fibres were alkali-treated to improve adhesion and reduce moisture absorption, and their mechanical properties were examined and optimized using Taguchi procedures based on grey analysis. The nanosilicon filler-based composite materials were built using a simple hand lay-up procedure.

2. Experimental Works

2.1. Materials. This study made use of a Dharbai fibre mat. Jevanthi Enterprises in Chennai, Tamil Nadu, supplies these fibres. Normally, the Dharbai fibre mat was produced in the following manner. The Dharbai leaves are cut off using a knife and dried for three days in the shade. The fibres were then water retted for eight days and washed to remove any foreign pollutants that had developed during the retting process. The fibres were then dried for two days at room temperature to eliminate the moisture content. These dried length fibres were then used to make the Dharbai fibre mat. The resulting fibres were treated with an alkali solution prepared by adding NaOH grains in freshwater. In this study, nano-silicon oxide powder is an essential substance used to reinforce Dharbai fibre. Aside from these fibres and reinforcing components, the chemicals employed in this study include epoxy resin, polyamine hardener, and distilled water. The nanopowder and other chemicals were procured from Naga Chemicals, Chennai, Tamil Nadu, India. Figure 1

Figure 1: Photographic image of reinforcement, filler, and matrix materials.

shows the photographic images of reinforcement, filler, and matrix.

2.2. Chemical Treatment of Natural Fibres. Dharbai fibres are pretreated for 4 hours using filtered water and a 5% alkaline solution that repairs the filaments' cracked surfaces and improves the filament's surface morphology. The filaments are rinsed in acetic acid to remove excess base until the pH on litmus paper approaches [21].

2.3. Sonication Process. By sonication, different weight compositions of nanosilica were employed to prepare a solution. 100 ml methanol was combined with three grammes of silicon material. After this combination was generated, sonication was initiated and lasted 2 hours. To avoid evaporation, the water bath temperature was kept below 25 degrees Celsius throughout the procedure [22]. The sonicated solution then was combined with 250 millilitres of epoxy at 45°C to evaporate the methanol, and constant swirling was done using the incubation equipment to ensure good blending [23]. The metal rod was used to ensure a precise mix, and the slurry was then prepared to plunge the fibres in. The binding agent had been added, and the solution was well stirred for 3–4 minutes to get a nice smoothness and uniformity [24].

2.4. Fabrication of Composites. To begin, a steel mould with dimensions of 150 × 150 × 3 mm was fine-tuned. The Nanosilica powder and woven Dharbai fibre composites were created using a manual lay-up method. The produced matrix solution was strewn across the layers of fibres in the mould. After the fibre mats were thoroughly soaked with matrix combinations, the mould was affixed and dehydrated in the open air for one day [25]. For three restrictions, each with three stages, the L9 orthogonal array was chosen according to Taguchi's design, and nine composite plates were constructed for further exploration. Desiccators were utilized to prevent the hybrid composite samples from absorbing moisture [26]. The parameters and their values are listed in Table 1. Table 2 reveals the L9 orthogonal array along with the ILSS outcome.

2.5. Cryogenic Treatment. The cryogenic procedures were carried out in a temperature-controlled cryogenic chamber with programmed controls. A regulated cooling rate (3°C/min) was used to get the temperature down to 196°C. The produced samples were submerged in liquid N_2 at 77 K for cryogenic treatment for 15 minutes, 30 minutes, and 40 minutes, respectively, according to the experimental design [27]. After the treatment, the composites were reheated at a continuous rate of 45°C/h to return to room temperature.

2.6. Testing. The fabricated composite samples were cut and rendered to the ASTM standard of D 2344 replicas with a dimension of 45 × 3 × 3 mm for interlaminar shear strength of the biobased nanocomposites.

2.7. Fractographic Study. SEM was utilized to conduct microscopic investigations into fractured composite samples [28]. The specimens were laved, dehydrated, and surface coated with 10 nm of gold before SEM clarity to increase the composites' electrical conductivity.

3. Taguchi Method

Taguchi's approach is the most effective for studying the relationship between various factors and executing fewer experimental plans, saving money and time. The mean result of quality features was used to perform the main effect evaluation at every variable range [29]. Furthermore, ANOVA was used to investigate the outcome and participation of each variable in the functional features [30].

3.1. Taguchi Investigation and Their Results. The Taguchi technique of the experimental process is a useful procedure for a logical explanation, research, and optimization of numerous process factors to achieve the desired effect. The investigative results are converted into an S/N ratio using this approach to discover the main constraints [31]. The S/N ratio characteristics were separated into three stages depending upon the conclusive reason for the major feature to be improved: (i) bigger the best, (ii) nominally the best, and (iii) smallest the best. The higher the S/N ratio, regardless of the performing feature group, the greater the performing feature [32]. As a result, the parameter's ideal level is the one with the largest S/N ratio. Flexural, tensile, and impact strengths were assessed throughout the composites testing using the greater principle [33]. As a result, S/N ratios of prominence characteristics (bigger is best) are explicitly expressed as [34]

$$\text{S/Nratio} = -10 \ \log 10 \ \frac{1}{e} \sum_{a=i}^{e} \frac{1}{X_{ab}^2}. \quad (1)$$

Table 1: Constrains and their stages for composite.

Sl. No	Parameters	Symbols	Levels		
			L1	L2	L3
1	Silicon powder (wt.%)	A	2	4	6
2	Woven Dharbai thickness (gsm)	B	200	250	300
3	Cryogenic treatment (min)	C	15	30	45

Table 2: L9 orthogonal array and experimental results.

Trail No.	Nano silicon powder (wt.%) A	Woven Dharbai thickness (gsm) B	Cryogenic treatment (No.) C	ILSS outcome (MPa)	S/N ratio of ILSS
1	2	200	15	35.81	31.0801
2	2	250	30	36.16	31.1646
3	2	300	45	34.59	30.7790
4	4	200	30	37.37	31.4505
5	4	250	45	35.32	30.9604
6	4	300	15	34.32	30.7109
7	6	200	45	34.10	30.6551
8	6	250	15	34.92	30.8615
9	6	300	30	35.34	30.9653

Table 3: Average values of S/N ratio of nanocomposites.

Sl. No	Constraints	Level 1	Level 2	Level 3
1	Nano silicon powder (wt.%)	35.52	35.67	34.78
2	Woven Dharbai type (gsm)	34.75	35.46	35.76
3	Cryogenic treatment (min)	35.01	34.67	30.06

Table 4: Mean value of ILSS outcome.

Levels	Nanosilicon powder (wt.%)	Woven Dharbai type (gsm)	Cryogenic treatment (min)
1	35.52	35.76	35.02
2	35.67	35.47	36.29
3	34.79	34.75	34.67
Delta	0.88	1.01	1.62
Rank	3	2	1

Table 5: S/N values of ILSS outcome.

Levels	Nano silicon powder (wt.%)	Woven Dharbai type (gsm)	Cryogenic treatment (min)
1	31.01	31.06	30.88
2	31.04	31	31.19
3	30.83	30.82	30.80
Delta	0.21	0.24	0.40
Rank	3	2	1

Table 2 shows the results of interlaminar shear strength and associated S/N ratios. The mean data for S/N ratios may be computed using the data in Table 2. The following approach can be used to determine average values [35], for example, the parameters of the silicon nanofiller.

$$\text{Level 1} = \frac{35.81 + 36.16 + 34.59}{3} = 35.52, \tag{2}$$

$$\text{Level 2} = \frac{37.37 + 35.32 + 34.32}{3} = 35.67, \tag{3}$$

$$\text{Level 3} = \frac{34.10 + 34.92 + 35.34}{3} = 34.78. \tag{4}$$

The finest ideal parametric levels were discovered utilizing an ANOVA table, including stage 2 from silicon filler (4 wt.%), stage 1 from woven Dharbai type (200 gsm), and stage 2 from cryogenic treatment (30 min).

3.2. ILSS Properties of Nanocomposites. The interlaminar shear strength of the composite frame was tested using a universal testing machine (UTM) with a load cell limit of 5 kN. The sample was cut according to ASTM standard D-2344. A 45 mm long bar with a minimum thickness of (width = thickness) and square cross-segments was loaded under three-point bending [36]. The ILSS response of composites is used to detect if the material has shear behaviour between its layers. On composites, the ILSS test assesses

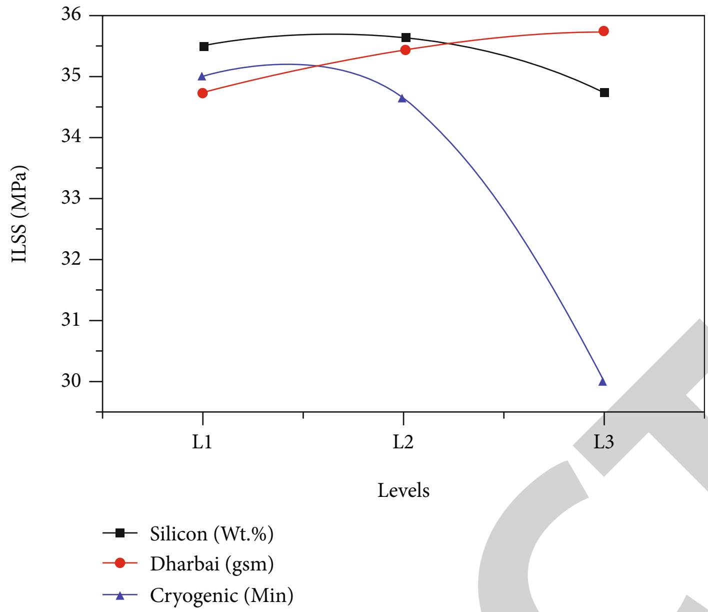
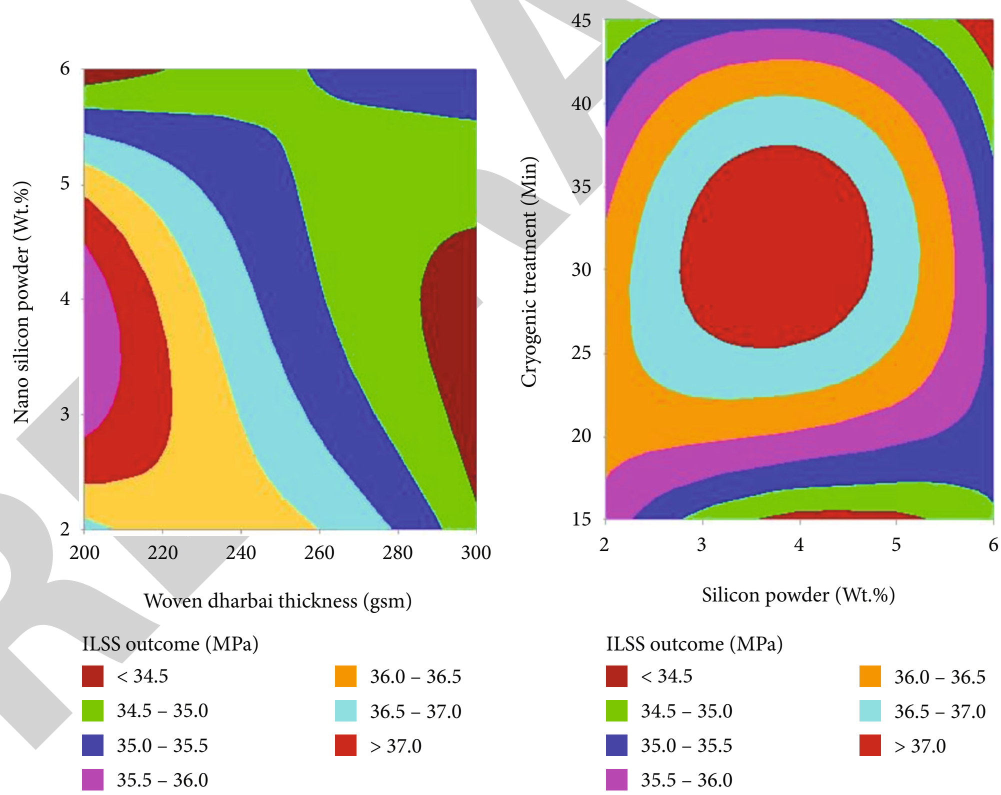


FIGURE 2: ILSS behaviour of nanocomposites under cryogenic environment.



(a) (b)

FIGURE 3: Contour plots of nanocomposites: (a) nanosilicon vs. Dharbai mat; (b) nanosilicon vs. cryogenic treatment.

the bonding between layers to resist shear pressure at a specific location [37]. Table 3 shows the average values of the S/N ratio of nanocomposites. The mean value of ILSS outcome is shown in Table 4, and the S/N values of ILSS outcome are shown in Table 5. The ILSS response of composites is used to determine if there are shear behaviours between the layers of the material. The ILSS examination is performed on composites to determine the bonding between layers to resist shear pressure at a specified spot. The bending modulus and deformation between the plies strongly rely on the

Table 6: ANOVA for ILSS strength of nanocomposites.

Constraints	DOF	SOS value	Variance	Percentage of influence
Nano silicon powder (wt.%)	2	1.341	0.6703	18.30
Woven Dharbai type (gsm)	2	1.620	0.8099	22.11
Number of layers (No.)	2	4.366	2.1830	59.58
Total	6	7.327	3.6632	100

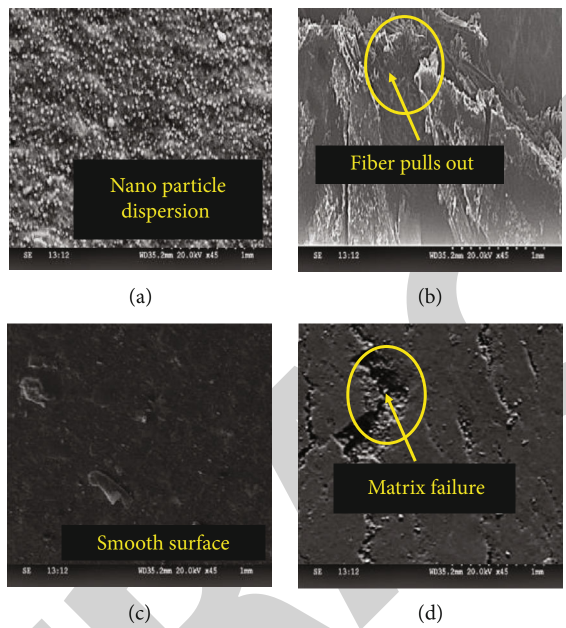


Figure 4: Microstructural images of nanocomposites under a cryogenic environment.

delamination between layers [38]. If the load is applied on one ply, the failure between the composite layers results in complete failure. This failure is assessed through the ILSS testing. Generally, the higher value of ILSS indicates the strong adhesions between nanofiller, epoxy matrix, and Dharbai fibres [26, 27].

Compared to the first and third levels (such as 2 and 6 wt.% silicon), the second levels (such as 4 wt.% silicon) produce the highest ILSS values in Figure 2. Increasing the nanosilicon content in composites enhances the interlaminar shear strength. However, ILSS was particularly high in 4 wt% of silicon [39]. More silicon in the matrix leads to improved matrix bonding, which results in a stronger reinforcing effect. Because of the functional groups on the nano-silicon oxide interface, the degree of cross-linking in the specimens rises, enhancing the shear behaviour. But the above results are suitable for only up to 4 wt.%. When the silicon powder increases beyond those levels, it lowers the mechanical strength [40]. This may happen due to the improper dispersion of silica particles in the epoxy matrix [41]. In the case of Dharbai fibre, all the levels provide positive effects. It demonstrates that thicker woven Dharbai (i.e., 300 gsm) would improve nanocomposite strength. It is because the presence of thicker, higher-density fibre materials reduces the impacts of voids, which is consistent with authors Natrayan and Merneedi, Velmurugan and Kaliyamoorthy, and Islam et al.'s findings [3, 20, 25]. Cryogenic treatment of polymer-based composites is a novel method of improving mechanical characteristics. The consequence of cryogenic handling on the mechanical characteristics of polymer-based hybrid composites is depicted in Figure 2. Compared to the second and third levels (30 and 45 minutes), the first level (15 min) provides the highest mechanical strength [42]. It might be owing to the residual stress created by the compressive interface due to the cryogenic exertion of composite materials. Because of the changing matrix and fibre shrinkage, residual stresses are created at low temperatures. The interface mentioned above stresses assists in keeping fibre and matrix in contact and enhance adhesion, resulting in better outcomes. When the composites are treated beyond this limit, it can create negative effects like matrix cracking, potholing, and misalignment of fibre orientations. This may happen due to the formation of higher compressive stress at the boundary surface. Figures 3(a) and 3(b) show the contour plots of nanocomposites at various parameters.

3.3. ANOVA. The ANOVA results are utilized to determine which process characteristics influence the impacting process variables of nanosilicon and Dharbai-based hybrid composites. The ANOVA may be written using the common outcome values. The percentage contribution through the total sum of the squared deviations may be used to assess the relevance of a processing parameter modification on concert attributes.

Table 6 shows the effect of each process parameter with nanosilica powder, woven Dharbai, and the cryogenic treatment contributing 18.30%, 22.11%, and 59.58%, respectively. This suggests that the cryogenic treatment is the most important characteristic of the silicon-based nanocomposite.

3.4. Fractographic Analysis. The nanocomposite morphologies at the fractured interface (following ILSS) were examined using SEM (Carl Zeiss, Supra 55). The photos support previous findings that the composites had lower permeability due to consistent fibre dispersion. The silica particles on the epoxy matrix are shown in Figure 4(a). Pull-outs of fibres and fibre packs result in the holes shown in Figure 4(b). The SEM test indicated that the matrix and reinforcement had a good connection. The resin, for example, has saturated all of the reinforcements. It is further said that the fibres in the resin system are well distributed. Figures 4(c) and 4(d) show the specimen after 30 and 45 minutes of cryogenic storage. It could be due to residual stress created by the compressive interface due to composite materials' cryogenic exertion. At low temperatures, residual stresses are formed due to changing matrix and fibre shrinkage. This interfacial stress aids in the contact and adherence of fibres and matrices, resulting in good consequences [30, 31]. The mechanical characteristics of composite specimens degraded when exposed for longer than 30 minutes. Longer cryoprocessing periods may result in higher thermal stress because of the increasing quantity of fabric mismatch. Because deformation is driven by decreasing contact, it is a far more dangerous composite structure. It might be because the time it takes to cure the epoxy resins is significantly decreased.

4. Conclusion

The interlaminar shear strength of silicon and Dharbai-based hybrid nanocomposites was examined in the present study, and the Taguchi technique was used to optimize the process parameters. The following findings are the results of this experimental study activity:

(i) As per the Taguchi analysis, the suggested stages of nanosilicon filler and Dharbai-based hybrid composites are 4% weight ratios of silicon powder and 300 gsm of Dharbai fibre mat and 15 minutes of cryogenic treatment. The combinations mentioned above provide the highest strength of ILSS

(ii) Nanoparticles and cryogenic treatments provide a high amount of residual stress in the composite interface, which helps to increase the interfacial bonding between the reinforcement and the matrix

(iii) According to the ANOVA, the cryogenic treatment was the most significant factor, contributing up to 59.58%, followed by woven Dharbai mate, contributing 22.11%, and nanosilicon at 18.30%.

(iv) SEM images demonstrate the interfacial bonding between the reinforcement and the epoxy matrix under cryogenic conditions

Data Availability

The data used to support the findings of this study are included within 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.

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