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Research Article

Influence of Nanosilica Particle Addition on Mechanical and Water Retention Properties of Natural Flax- and Sisal-Based Hybrid Nanocomposites under NaOH Conditions

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Organic filament-based lightweight materials are increasingly being used because of their high strength-to-weight ratio, recyclability, and low cost. The application of nanofillers in addition to natural fibres is a fascinating one. The main purpose of the current experimental investigation is to manufacture and estimate the mechanical material of nanocomposites. Natural fibres like flax and sisal are used as reinforcement; nanosilica particles act as fillers, and epoxy resin as a matrix. The composites were created using the Taguchi L₉ orthogonal array and a hand lay-up technique. The mechanical and water retention behaviour of the hybrid composites is based on the following three parameters, each with three different levels: (i) adding different weight ratios of nanofiller (1.5, 3, and 4.5 wt%), (ii) weight ratio of reinforcements (20, 30, and 40 wt%), and (iii) duration of NaOCl conditions (2, 4, and 6 hours). Mechanical possessions like tension, bending, and impact were tested as per the ASTM standard. The tested composites show that 30 wt% reinforcement, 3 wt% nanosilica, and 4 hours of alkaline processing provide the best materials and aquatic preoccupation belongings. When compared to nanofiller composites, nanoparticle-filled composites have 17% evolution in tension, 22% upsurge in flexural strength, 13% in impact strength, and 36% increase in impact strength hygroscopic behaviour. Scanning electron microscopes were used to analyze the fractured structure of hybrid composites. Compared to 1.5 and 4.5 wt% of nanofiller, the 3 wt% of filler provides high interfacial adhesion to the hybrid composites. It helps the reinforcement and matrix to contact each other.

1. Introduction

Polymeric materials have seen tremendous growth in recent years with the use in sports goods, internal structures, automobile interiors, electrical and mechanical products, aerospace enterprises, and household goods, among other things. Low cost, easily accessible, high tensile strength to weight ratio, resistance to abrasion, extended fatigue resistance, and great renewability are just a few examples [1]. Polymer composites have a lengthy history of design and analysis, which numerous Nobel Prize winners have furthered over the last century by changing different factors to build new and durable

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combinations [2, 3]. Due to its distinctive qualities, flax fibre is perhaps the essential member of the bast group for composite reinforcement. Bast fibres are extracted from fibrous strands found within the root bark of a plant stem [4]. Flax fibre's intrinsic high-load carrying capacity and its minimal extension to breakdown are essential qualities that make it appealing for composite development. Flax (Linum usitatissimum) is commonly grown in areas with great weather. Flax plants are frequently farmed in Europe, according to Charlet et al. [5] where the average temperature is normally around 30°C. Flax is produced in Southern Europe, Brazil, Pakistan, Russia, and Columbia, among other places. Flax fibres, unlike synthetic fibres, are not unbroken but have a composition comparable to composites and are hierarchically arranged [6]. The micro- and nanostructural structure of the economy determines its macroeconomic qualities. Flax has been utilized as a significant industrial fibre since prehistoric times. Ancient explorers used braided natural flax fibres to make ropes for hafting primitive tools, making baskets, and stitching clothes over 30,000 years ago [7]. The mechanical characteristics of flax fibres were shown to be controlled by their placement as in stems by Charlet et al. [8]. The noncellulosic materials are the matrix constituents that facilitate the transfer of stress through one microfilament to another, whereas cellulose is the comparable binding material of a composite structure. Because of the similar composite-like nature of this fibre, Bos et al. [9] found that the flax fibre strength diminishes as the restraining duration rises. They used tensile experiments to compare the toughness of fundamental and technological flax fibres and discovered that owing to a clustering effect, fundamental flax fibres had significantly higher toughness than technological fibres of the same lengths [10].

A mixed composite is made up of many numerous ferromagnetic layers in a single material. By eliminating the drawbacks of individual combinations, hybridization can enhance the properties of organic fibre-reinforced polymeric materials [11, 12]. Sisal plants are members of the Agavaceae family, and their scientific name is Agave sisalana. The fibre is taken from the leaves of various plants. The fibre's nature and qualities differ depending on where it is grown. Although the origins of such plants are uncertain, they are endemic to America's southern region [13]. However, these plants are grown for their fibres all over the globe. Argentina is the world's greatest grower of such species. Some plants may even thrive in arid environments with little water. Sisal fibre plant growth accounts for 2% of all planting globally. Throughout their lives, sisal fibre plants generate 250-300 leaves. Every drying leaf contains 4% of its full weight in fibres. A sisal plant leaf weighing roughly 650 g has 200 to 1000 fibres. The diametre ranges from 200 to 500 mm. The fibres were removed in bundles with lengths ranging from 0.5 to 1.0 metres. Every sisal plant leaf contains an average of 80-88 percent water, 3-5 percent fibre, 0.5-0.8 percent cuticle, and 7-9 percent additional dry components [14]. Mechanical and manual extraction methods were used to separate the fibre from the plant. The retting and boiling processes are part of the manual extraction procedure. The fibre content changes regardless of the type of extraction method used.

Natural fibres have varied potential due to differences in cellulose, hemicellulose, and lignin chemical constituents [15]. The fibre and matrix must have interaction and affinities for the composite to have superior attributes. Flax and sisal fibres are lignocellulosic fibres with OH compounds that absorb water fast and degrade the performance of natural composites, notably their high stability in their dimensions. Natural fibres do not cling well to nonpolar matrices because of their polar functional groups. Natural fibres are chemically pretreated to eliminate this intricacy [16]. Chemically pretreated fibre surfaces reduce hydrophilicity, improve mechanical properties, and increase thermostability by lowering the concentration of hemicelluloses, lignin, and waxes on the fibre. Sodium hydroxide is a common chemical that alters the boundaries between incompatible materials like raw fibres and resin. NaOCl removes the usual wax and glosses off the cellulose fibres externally, activating the process's hydroxyl group. In the same way, NaOCl reacts with nearby OH bonds in fibres and removes cellulose, water, and contaminants in the same way [17].

However, polymer composites have several limitations, including difficult manufacture, fibre debonding, limited damage tolerance, poor stability, low elasticity, and low hardness which limit their use to reduced material [18]. The use of nanosized materials to create polymeric-based nanomaterials is presently advanced to close the gap between polymeric material services and technical specifications. Nanocomposites are compositions created by combining a polymer matrix with a nanoparticle dispersion [19, 20]. The addition of nanoparticles to polymeric materials has been used to improve a variety of qualities, including improved hardness and mechanical properties, great thermoelectric properties, remarkable heat resistance, and a much more significant barrier to water or hydrocarbons [21]. Many researchers have examined the consequence of silica nanoparticles on the physicochemical, rheological, dynamic, and thermal belongings of artificial fibre strengthened polymer composites. Although artificial resourced polymeric materials outperform natural fibre-based polymer composites, their environmental acceptability is constantly questioned. As a result, natural fibres are increasingly being used to replace traditional synthetic fibres in constructing polymeric materials. Puttegowda et al. [22] found that phenol nanocomposites with kenaf and PALF hybridization had improved thermodynamic and static mechanical characteristics. It has been found that combining kenaf with pineapple increases the material and aquatic fascination belongings of polyethylene compound constituents. Jiang et al. [23] hybridizing Prosopis-carbon-fibreglass and kenaf-nanofiller-Kevlar filaments strengthened syntactic materials and improved mechanical performance. Chee et al. [24] showed that incorporating silicon filler with magnetite materials and a timber floor improved the materials' characteristics. Singh et al. [25] examined the physical and heat resistance features of kenaf-flax strengthened epoxy hybrid nanocomposites after adding nanoclay and halloysite nanotubes. They discovered that adding montmorillonite nanoclay to hybrid nanocomposites boosted densities while lowering void content and water retention, whereas



FIGURE 1: Flax fibre abstraction from flaxseeds shrub.

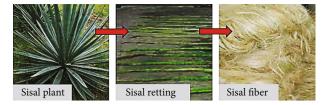


FIGURE 2: Sisal fibre abstraction from Agave sisalana shrub.

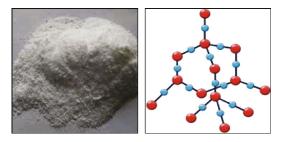


FIGURE 3: Photographic image of nanosilica powder and their chemical structure.

biopolymer montmorillonite nanoclay produced nanomaterials with hyperdimensional integrity [26].

The main purpose of the present investigation is to build and test the mechanical and water retention belongings of hybrid organic nanocomposites utilizing the following criteria: nanosilica particle concentration, flax and sisal weight ratio, and NaOCl treatment hours. The nanosilica fillerbased composite materials were made using a simple hand lay-up procedure. Alkali was used to enhance adhesion and reduce hygroscopic in natural fibres.

2. Experimental Works

2.1. Resources. The flaxseeds and sisal fibre reinforcing materials were obtained through Globe Fiber Industry, India. The fibres were carefully cleaned with sparkling water and sundried for 48 hrs to eliminate the moisture. Figure 1 depicts the abstraction of flaxseeds strengthening elements from its shrubberies. Figure 2 shows the sisal fibre abstraction from Agave sisalana shrub. This research used nanosilicon–oxide particles and an epoxy matrix. The matrix and nanofillers were procured from Rithu Chemicals, India. Figure 3 illustrates the detailed image of silica and its chemical structure.

Table 1: Limitations and their stages of hybrid nanocomposite.

Sl no.	Limitations	Signs	Levels		
			L1	L2	L3
1	Nanosilica content (wt%)	wt%) A		3	4.5
2	Weight % of flax and sisal (wt%)	В	20	30	40
3	NaOCl treatment (hrs)	C	2	4	6

Table 2: L₉ orthogonal array of hybrid nanocomposites.

Trail no.	Nanosilica content (wt%) A	Weight % of flax and sisal (wt%) B	NaOCl treatment (hrs) C
1	1.5	20	2
2	1.5	30	4
3	1.5	40	6
4	3	20	4
5	3	30	6
6	3	40	2
7	4.5	20	6
8	4.5	30	2
9	4.5	40	4

2.2. Alkaline Processing. The water retting procedure was used to remove fibre from the bark of flax and sisal. Both strands were processed with a 5% alkali solvent in the trays. The fibres were steeped in the mixture for 4 hours for the best results. The soaked fibres were thoroughly washed after removing them from the mixture to eliminate any remaining alkaline solution. A final wash was performed using distilled water to clean the entire surface. The fibres were then dried out in a furnace at 60°C for different hours like 2, 4, and 6 hrs to remove any remaining humidity.

Mercerization is an alkali treatment method. It is widely used in the clothing industry. According to ASTM D1965, recrystallization exposes a vegetable fibre to a suitably saturated solution of a suitable platform resulting in significant expansion and changes in fine structure, shape, morphology, and mechanical behaviour. Natural fibre alkali treatment is a chemical deposition method that modifies the chemical behaviour of natural fibre components. Introducing sodium hypochlorite (NaOCl) to natural fibre enhances hydroxyl ionization to an aldehyde group. The consequence of NaOCl on lignocellulose is a stretch response in which the ordinary crystal assembly of roughage fractures. As a consequence of alkali treatment, the corresponding reactions take place.

Vegetable fibre – OH + NaOCl
$$\longrightarrow$$
 Fiber – O – Na + H₂O. (1)

2.3. Preparation of Nanocomposites. In the first step, nanosilica plus resins were blended using a motorized spinning technique for 15 minutes to combine the matrix and the added substances. The ultrasonicator is used to spread its fillers into matrices using Doppler ultrasound. Various weight combinations of nanosilica weight percentages were

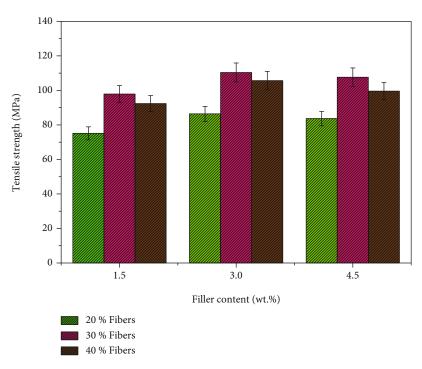


FIGURE 4: Tensile strength of nanocomposites with different filler and fibre content.

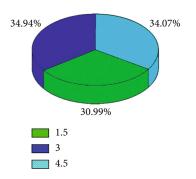


FIGURE 5: The contribution of silica filler in terms of weight percentages to tensile strength.

employed to create a nanostructure, including 1.5, 3, and 4.5 wt%. The submicron silicon and resin combination were mechanically stirred in a glassware beaker and held in an enhanced ultrasound cleaner in pulse mode for about 45 minutes. A $150 \times 150 \times 3$ mm steel mould was used to create the nanocomposites. The composite lamination wax was first applied to a mould to make it easier to separate it. Matrices are a 10:1 combination of resin and curing agent. The mixture was then filled with fillers ranging in weight from 1.5 to 4.5 grammes and equal dimensions (micro). The solution was continuously spun for 10 minutes to achieve thorough mixing. In this scenario, 40% of the solution was poured into the mould first followed by the placement of treated (oven-dried) fibres. Finally, the remaining matrices were poured over the fibres. Using a roller, the grid was equally applied to all four borders. To obtain constant lamination width and remove additional matrices from the

mould, 12 kg of tension was retained on the mould resulting in a 3 mm composite with a limited dimension. To properly dry the constructed laminate, the mould was placed in a 75 $^{\circ}$ C microwave oven for 3 hours. After that, the lamination was split into parts and tested according to ASTM standards. Table 1 list the parameter and the levels of the nanocomposites. Table 2 revealed the L₉ orthogonal array of nanocomposites based on their parameters.

2.4. Composite Testing. For the tension test, the produced laminate sections were characterized and converted to the ASTM specifications of D 638-03; ASTM D-2344 for ILSS and D-790 for bending. The following equations were used to find the mechanical tensile and flexural strength:

Tensile strength =
$$\frac{P}{b*t}$$
, (2)

where P = applied load, b = width, and t = thickness,

Flexural strength =
$$\frac{3PL}{2hd^2}$$
. (3)

- 2.5. Microstructural Analysis. Morphological examination of cracked laminate assays was carried out using SEM. Before SEM examination, all materials are laved, drained, and externally encased using tens of nanometres of precious metals to improve the ionic properties of compounds.
- 2.6. Water Retention Behaviour. The mixture of composite materials produced rectangular samples measuring 39 mm \times 10 mm \times 3 mm. The examples were microwave dried for 1 hour at 80°C then cooled to a constant weight outside.

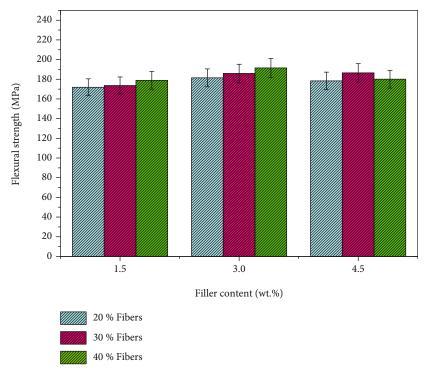


FIGURE 6: Flexural strength of nanocomposites with different filler and fibre content.

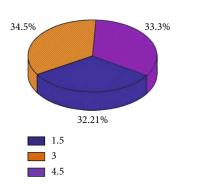


FIGURE 7: The contribution of silica filler in terms of weight percentages to flexural strength.

The composite models were then occupied in sanitized aquatic for 10 days as required by ASTM D570. The models were removed from the moisture daily, cleaned with tissue paper, reweighed and quantified, and then returned to the liquid. The water uptake rates were calculated using the following formula:

Moisture absorption =
$$\frac{W_2 - W_1}{W_1} \times 100$$
. (4)

 W_2 is the weightiness of the model after immersing, and W_1 is the weightiness of the model before immersion. Each sample was subjected to five experiments with the average results provided.

3. Results

3.1. Tensile Strength of Nanocomposites. Three specimens of each composite construction measuring $150 \times 25 \times 3$ mm were cut according to ASTM requirements. The maximal strength was calculated from the test report chart after the strength was recorded on a UTM. Figure 4 depicts the results obtained. The mean and standard deviation were calculated using the three sample results and are shown as error ranges. Figure 4 indicates that increasing the insoluble fibre up to 30% by weight enhances tensile strength while increasing the fibre percentage beyond that reduces tensile modulus. It results from defective matrices creating inappropriate matrix-fibre binding; as the number of reinforcements increased, the matrix number decreased [27]. Previous studies in the same domain have produced similar results.

The current research is unusual because it looks at three different filler materials separately. According to the uniaxial tensile findings, a composite with 3 wt% nanosilica had a higher tensile strength (110.36 MPa) with 30 wt% reinforcement than a composite without filled 30 wt% reinforcement. Figure 4 shows that the strength qualities have improved by 12%. It shows that the silica nanoparticles were efficiently disseminated in the epoxy matrix mixture compared to nanofiller composites [28]. The nanosilica fillers were employed which resulted in a greater interface adhesion between the matrix and reinforcement allowing for stressstrain transfer. Figure 4 shows how adding filler materials improved the tensile strength. The contribution of silica filler in weight percentages to tensile strength is shown in Figure 5. As a result, epoxy formulations with 30 weight% reinforcement provide adequate grip obligatory among superficial bonds at an attentiveness of 3 weight%. In

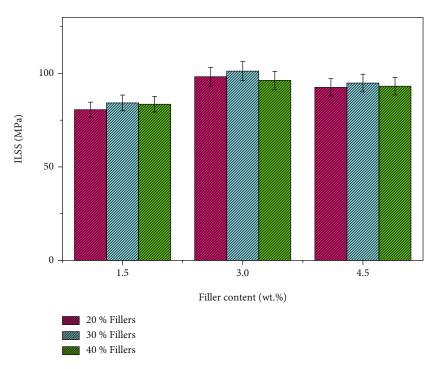


FIGURE 8: ILSS of nanocomposites with different filler and fibre content.

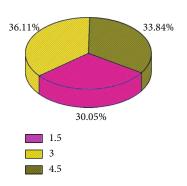


FIGURE 9: The contribution of silica filler in terms of weight percentages to ILSS.

dissimilarity, accumulation of 1.5 and 4.5 weightiness% nanosilica caused an undesirable consequence representing a drop in materials strength. Moreover, the interface attachment of fibre and matrix in reinforcement materials has been proven weak at 1.5 wt% and 4.5 wt%, subsequent conglomeration owing to poor adherence and inferior nanocomposite métier characteristics [29].

3.2. Flexural Strength. The bending strength of the produced composite samples was tested using ASTM standards. The specimens were made from laminated composites with dimensions of $150 \times 12.7 \times 3$ mm [30]. Three distinct instances of the same laminate were cut and evaluated for uniformity. Figure 6 demonstrates that the laminate sample's flexural strength increased up to 30 weight% but that as the fibre content increased, flexural strength decreased comparable to tensile strength [31]. Several detectives described similar conclusions. The influence of fillers on flexural strength was also investigated [32] with 3 wt%

nanosilica-filled 30% reinforced composites, and the greatest flexural strength of 191.58 MPa was reached similar to tensile strength [33]. Figure 6 illustrates that silica as a filler material improves flexural strength by 16% compared to empty flax and sisal fibre reinforced homogeneous mixture [34]. All additive materials improved flexural strength compared to the empty sample material. Adding fillers improved the matrix and fibre's load-sharing capacities [35]. The fillers improve matrix-to-reinforcement adhesion resulting in a considerable improvement in dynamic load capacity from the reinforcements to the matrix [36]. The contribution of silica filler in weight percentages to flexural strength is shown in Figure 7.

3.3. Interlaminar Shear Strength. Composites' ILSS response determines whether the material exhibits shearing behaviour among its layers. The ILSS test is accomplished on composites to evaluate layer bonding to withstand shear pressure at a specified point [37]. The second levels (such as 3 wt% silicon) provide the greatest ILSS values when compared to the first and third levels (such as 1.5 and 4.5 wt% silicon) [38]. Increasing the amount of nanosilicon in composites improves interlaminar shear strength. However, in 3 wt% of silicon, ILSS was shown to be exceptionally high (101.25 MPa) [39]. High silicon content in the matrix improves matrix bonding resulting in greater strength properties shown in Figure 8. The amount of cross-linking in the samples increases due to the functional groups on the nanosilicon-oxide interface, which improves the shear behaviour [40]. However, the initial results are only valid up to a weight of 3%. The mechanical strength decreases when the silicon powder concentration rises above such levels. It might be caused by poor silica particle dispersion in the epoxy matrix [41]. Flax and sisal concentrations are beneficial in flax and

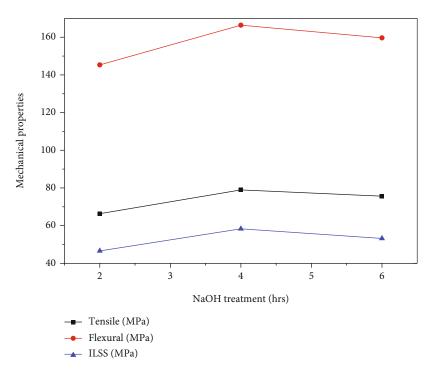


FIGURE 10: Mechanical properties of nanocomposites based on the NaOH treatment hours.

sisal combos. It reveals that the 30 wt% reinforcement successfully transfers the load to the matrix compared to 20 and 40 wt% reinforcement [42]. It might be owing to the epoxy matrix's sufficient adhesive bonding [43]. The contribution of silica filler in weight percentages to ILSS is shown in Figure 9.

3.4. Effect of NaOCl Treatment. Figure 10 represents the experiments revealing that a 5% alkaline solution concentration and a 4 hour soaking time increase mechanical performance. This fault could be caused by poor fibre absorption at sodium hydroxide levels of more than 4 hours [44]. Using a 5% proportion of alkaline solutions over a 2-4 hour period yields the highest ILSS, flexural, and other properties [45]. The filaments that had been processed for six hours were extremely strong. On the other hand, the bulk of the substances handled with them lost strength. This is because fibre treatment degrades fibres across the polymers by altering the cell structure of the fibre over a long period (>4 hours) [46]. The fibres become rigid and brittle due to the crystalline growth resulting in increased resistivity and poor extensibility. Due to their enhanced fragility, these textiles shrunk even more when pressured and they could not efficiently transmit load at interfaces decreasing the material's properties. This was confirmed by photographs taken with a scanning electron microscope (SEM). Most nine composite plates show favourable mechanical features only in the 2 to 4 hour NaOCl treatment zones, particularly in the 4 hr sections.

3.5. Water Retention Behaviour. Figures 11(a)-11(c) show the proportion of water absorbed by flax and sisal fibre

reinforced hybrid composites after NaOCl treatment (2 hours, 4 hours, and 6 hours). The Indians' concern with water has changed dramatically over time. Fibre reinforcement induced high moisture retention in polymer composites in general. The relative humidity of 20 wt% and 40 wt% flax and sisal fibre-reinforced composite materials were low, but the moisture content of 30 wt% flax and sisal fibre-reinforced composite materials was high. The mechanical-fractured sample investigation revealed that 30% of fibre composites exhibited good fibre-to-matrix bonding capabilities. This primarily resulted in the development of fine water resistant properties. The polymer matrix is securely wrapped around the fibre due to excellent bonding, preventing water molecules from accessing the exterior. Due to the weak link between the matrix and the fibre, water molecules may easily access the fibre surface in the 20 wt% and 40 wt% fibre reinforced composites. The composite's water retention was increased as a result of this. After 250 hours of incubation, the 20 wt%, 30 wt%, and 40 wt% flax and sisal fibre reinforced composites achieved saturation point.

4. Microstructural Analysis

Figure 12 shows the SEM findings of untreated and treated failure samples of nanolaminated composites about temporal variation in fibre alkali processing. It is obvious that for fibres treated using NaOCl at a five colloidal solution for up to 4 hours, the tensile and flexural strength increase as the NaOCl treatment duration in the treating solution increases during the tensile and flexural strength decrease. Nevertheless, as the length of NaOCl exposure increased,

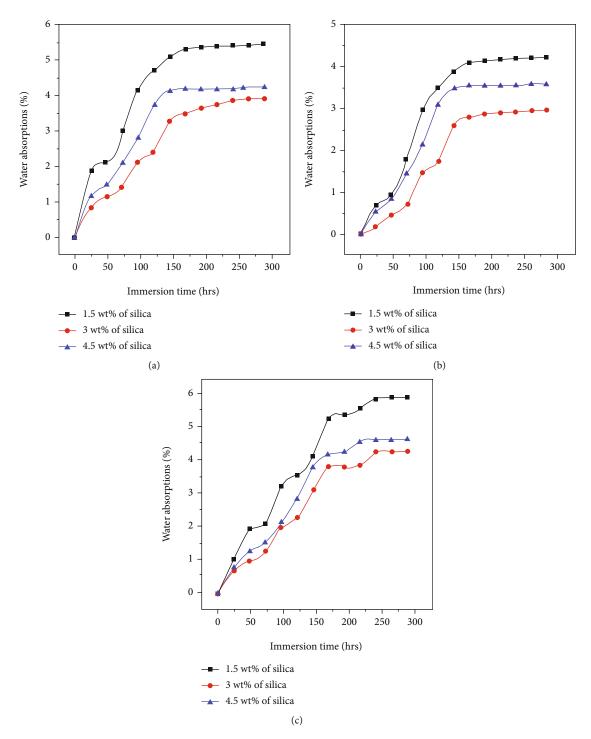


FIGURE 11: Water retention behaviour: (a) 2 hrs NaOCl, (b) 4 hrs NaOCl, and (c) 6 hrs NaOCl treatment.

the interlaminar shear strength increased linearly. Figure 12(a) shows that the quantity of hydrophilicity is lower due to the hydrophilic nature of the untreated fibres. As a result, the fibre–matrix contact is weak allowing for simple fibre withdrawal following failure as seen in Figure 12(b). However, the fibre geometry is altered due to the alkaline treatment and the fibres flatten. The SEM findings for failed samples generated from fibre treated

with 4 hours of NaOCl are shown in Figure 12(c). An interlaminar fracture is visible in the treated sample rather than a pull-out as in the untreated sample as shown in Figure 12(c). Furthermore, as previously stated, Figure 12(d) depicts fibre flattening due to excessive fibre treatment (6 hours). As a result of this overtreatment, the fibres become brittle. It was reducing the composites' mechanical characteristics.

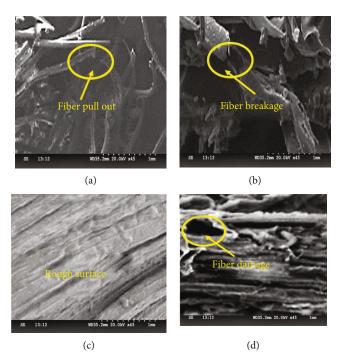


FIGURE 12: Microstructural analysis of (a) untreated, (b) 2 hrs NaOCl treated, (c) 4 hrs NaOCl treated, and (d) 6 hrs NaOCl treated fibres.

5. Conclusion

The hand lay-up technique effectively invented the flax/sisalbased hybrid nanosilica additions of bio materials and the following observations were made:

- (i) Among the various combinations, the 3 wt% of nanosilica, 30 wt% of flax and sisal fibres with 4 hours of NaOCl treatment provide the highest mechanical strength (110.36 MPa of tensile, 191.58 MPa of flexural, and 101.25 MPa of ILSS)
- (ii) Compared to 1.5 and 4.5 wt% of nanosilica, the 3 wt% of nanosilica inclusion exhibits the highest mechanical strength because 3 wt% of silica particles are thoroughly mixed with epoxy resin and show good bonding strength
- (iii) Maximum fibre pull-out occurs at 20 and 40 wt% of flax and sisal combined reinforcements. At the same time, 4 hrs of alkaline treatment effectively alter the fibre surface and provides good hygroscopic characteristics
- (iv) When compared to nanofiller composites, nanoparticle-filled composites have a 17% upsurge in tensile strength, a 22% upsurge in flexural strength, a 13% upsurge in impact strength, and a 36% increase in hygroscopic behaviour

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

According to the authors, there are no competing interests surrounding the publishing of this research.

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