Composites Science and Technology

Senthilkumar Krishnasamy ·
Mohit Hemath Kumar ·
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Suchart Siengchin *Editors*

Interfacial Bonding Characteristics in Natural Fiber Reinforced Polymer Composites

Fiber-matrix Interface In Biocomposites

Springer

Composites Science and Technology

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Introduction to Interfacial Bonding Characteristics of Natural Fiber-Reinforced Composites

V. Bhuvaneswari, D. Balaji, M. Ramesh, and N. Srinivasan

Abstract Interfacial bonding of natural fiber reinforced composites is important to be in contact with reinforcement and matrix at the time of processing of a composite which brings in to close bonding. The strength of the composites will be more if the Interfacial bonding is high. Variety of materials, chemical treatment, and pre-processing before making composites which increases the interfacial bonding between the natural fiber matrix. The selection of a proper binder for the suitable fiber along with the ideal pre-processing technique is the key determining factor of the interfacial bonding technique. So, in this chapter, various specifications of fiber and matrix bonding are discussed and analyzed with the interfacial bonding using SEM image.

Keywords Interfacial bonding · Natural fiber · SEM · Natural fiber reinforced composite

1 Introduction

At the fiber-matrix interface, two phases or components undergo a physical, mechanical, or chemical combination at the reaction or diffusion zone. Among the many elements that decide composite's physical characteristics, interfacial bonding among the fiber and matrix is crucial [1–3]. Mechanical interlocking, molecular attractive

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forces, and chemical bonds all play a role in strengthening the interfacial adhesion between the fiber and matrix. The hydrophobic polymers are not compatible with the hydrophilic plant fibers. The presence of many hydroxyl groups in plant fiber, in moreover to the sugar syrup and viscous stimulants in fiber, acts as an obstacle to fuse to the nonpolar polymer matrix, reducing the efficiency of the fiber's procedural response with the mixture [4–7]. Therefore, it is essential to alter the interface structure of fiber and water-unsolvable polymer in order to develop a sensible composite with robust interfacial bonding and effective natural load transport across the interface. Several techniques have been explored to enhance the compatibility and bonding among hemicelluloses particulate and hydrocarbon-based polymers [8–11]. These include physiological therapies (such as plasticizer discharge, solvothermal, corona, and serum therapies) and physic-chemical treatments (i.e. laser, UV bombardment) as well as chemical modifications [12–14].

Matrices utilized in the creation of plant fiber composites include carbohydrates, organic matter, glucans, chitin, keratin, cellulosic materials, urethane, poly (vinyl alcohol, polylactic acid (PLA), and soy-based resins. Because the reinforcements and matrices in biodegradable composites have similar polarities, the composites are more compatible with one another and have stronger interfacial adhesion than their synthetic polymer counterparts [15–17]. In order to prevent dimensional instability caused by fiber swelling and subsequent loss of interface integrity, it is necessary to treat the fibers' surfaces in a certain way. The composite's fiber and/or filler system is embedded in a matrix, which is typically a homogenous and monolithic material. It is uniform throughout and serves as a glue or cement to hold reinforcing materials together. Matrix materials are used to shield reinforcement fillers from damage and to transfer loads to them [18–20]. In contrast, the matrix resin keeps plant fibers in place, so they can withstand loads of force and improve composites' performance for next to nothing.

On the other hand, the interface conditions of a composite system are crucial to the cooperation between plant fibers and matrix. Properties of plant fiber polymer composites are primarily determined by interface conditions. This zone has a different thermal expansion rate than the rest of the material, so it behaves as a separation hurdle among those resources, which have fundamentally different physiological and toxin characteristics. If the interface of a composite is poor, the material's vibratory and thermodynamic characteristics may suffer greatly. Worse still, it was rumoured that the physical properties were subpar. Fortunately, the composites have been treated with a wide variety of compatibilizers to improve their physiological, tribological, and heat characteristics. Because of its low price, acid hydrolysis is the most popular method for treating plant fiber composites [21–25]. By doing so, it cleans the fiber surface of any non-cellulosic components while leaving it slightly rough, which improves interfacial adhesion. More functional groups can be promoted by using pairing entities and any substance used to stabilize blends of immiscible polymers to improve fiber/matrix interfacial adhesion [26–29].

Interfacial strength (IFS) between the fiber and the surrounding polymer matrix is often cited as the mechanism responsible for the improved quantitative effectiveness of fiber-reinforced composites. When comparing the efforts of different organizations, it can be challenging to use IFS because of the lack of uniformity in the methodologies used to develop them. The interfacial shear strength (IFSS) between a fiber and a matrix can be measured with a few different techniques [30, 31].

2 Interfacial Bonding of Natural Fiber Composites

The interfacial bonding between the reinforcing fiber and matrix is perhaps the most critical parameter in the development of fiber reinforced polymer composites. This is most influencing factor in determining the mechanical strengths and other characteristics of the composites, such as load transfer, hardness, water absorbing capacity, etc. [32]. The factors influencing the interfacial bonding of natural fiber reinforced composites are (i) inter diffusion, electrostatic adhesion, chemical reactions and mechanical interlocking, (ii) the incompatibility between fiber and matrix, which results in poor dispersion resin over the fiber, and (iii) Interfacial adhesion between matrix and filler are significantly improving the performance of the composites.

2.1 Abaca Fiber Composites

When used as strengthening investigators in composite materials, abaca fibers show tremendous promise. The mechanical properties and interfacial interaction of a prototype abaca fiber/epoxy composite were investigated that after the fibers were coated with 5, 10 and 15% of sodium hydroxide for two hrs. Crystallinity, tensile strength, and Young's modulus of treated abaca fibers were all higher than those of untreated fibers, and the treated fibers also showed enhanced interfacial shear capacity with an epoxy. The fiber's stiffness and potential use in composites were diminished by more intense alkali treatments. According to the findings, abaca fiber-reinforced polymer composites benefit greatly through mild alkali handlings (like 5 wt. % NaOH for 2 h) (Figs. 1, 2 and 3).

Abaca fibers undergo structural and chemical changes when treated with alkali, and these changes are amplified as the alkali concentration is raised. Two hours of immersion in 5–15 wt. % of NaOH solutions alters the inner configuration and exterior anatomy of abaca fibers by solubilizing hemicelluloses and lignin. Containing high crystalline phase and low fibrillation were observed in abaca fiber bundles that had been treated with 5 wt. % of NaOH, and these fibers exhibited remarkable interfacial adhesion with epoxy. When the concentration of NaOH was increased, the cellulose crystallinity was reduced, and the bundles of abaca fibers became fibrillated. Epoxy resin adhesion was poor because of changes in the fibers' surface chemistry. Abaca fibers' exterior characteristics and achievement were found to be greatly enhanced after being treated with low concentrations of alkali [33, 34].

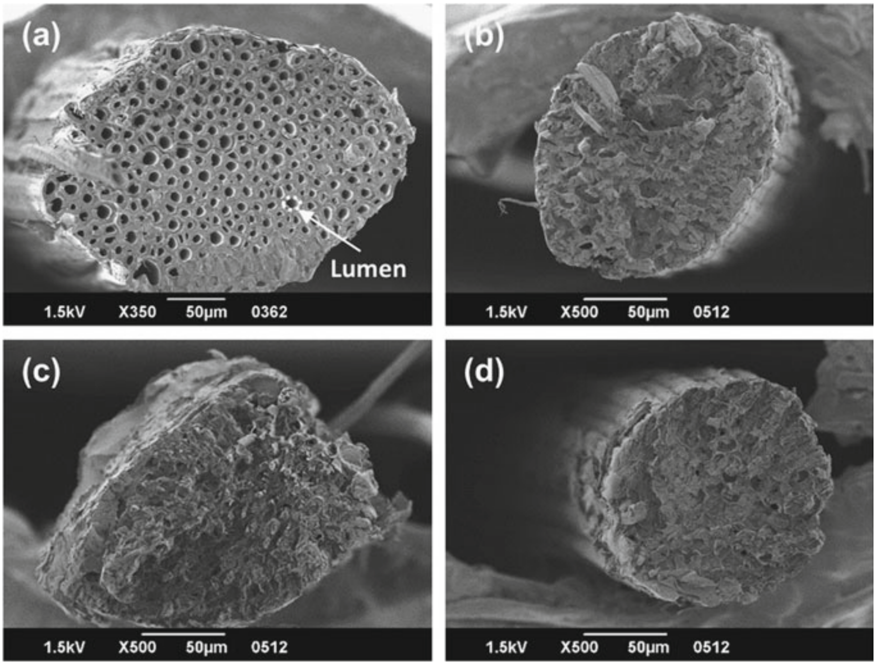

Fig. 1 SEM image of abaca fiber reinforced composite [33]

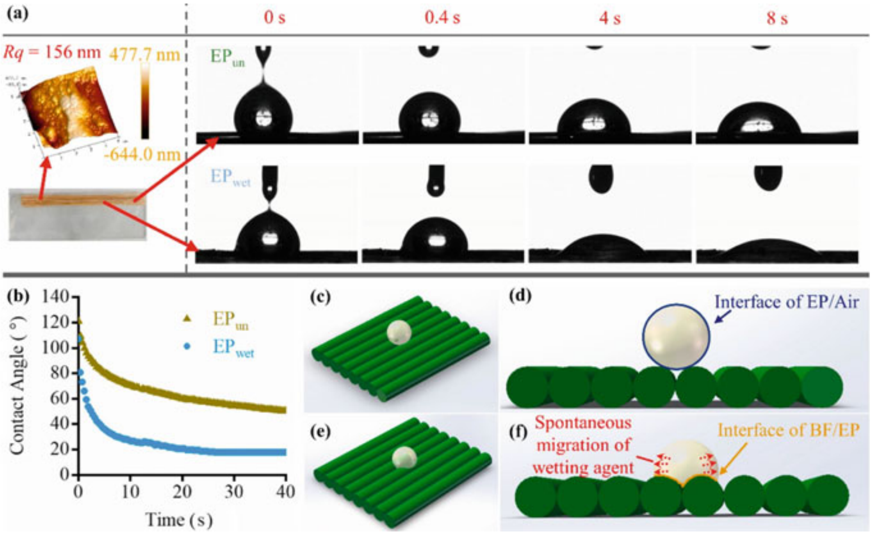

Fig. 2 Bamboo fiber bonding mechanism [35]

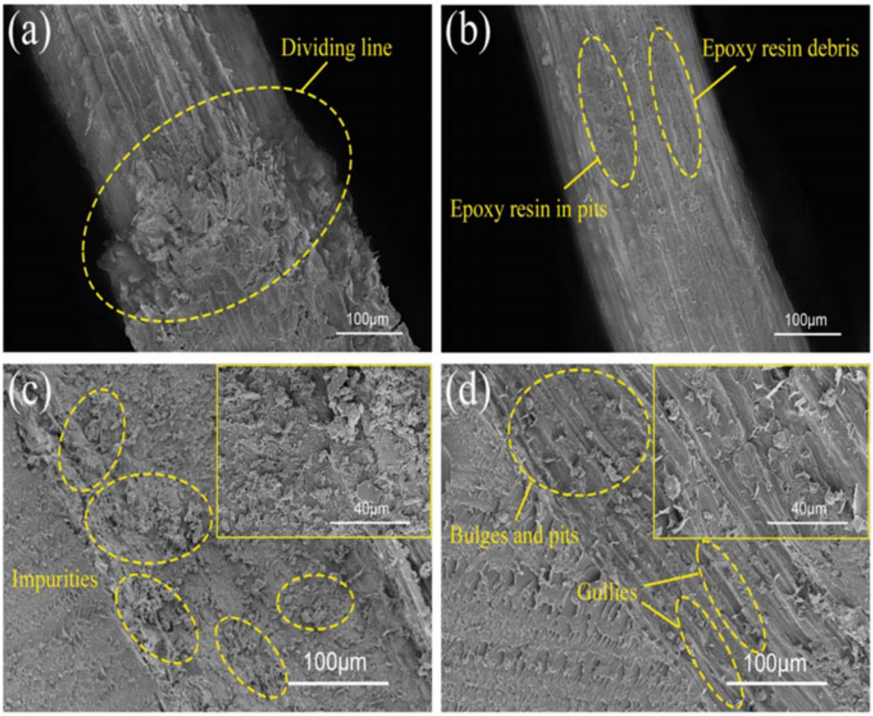

Fig. 3 SEM micrographs of **a** untreated coir fibers, **b** after alkali and silane treatment, **c** matrix of pull-out U-CFPS, and **d** matrix of pull-out A-S-CFPS [39]

2.2 *Bamboo Fiber Composites*

BYK-358N, a polyacrylate-based moisture operative, utilised here to strengthen adhesive interface among fibers of bamboo and matrix of epoxy. To investigate the curing of epoxy resin, researchers employed a Transformation function infrared spectrometer and a differential scanning calorimeter. Epoxy and bamboo fiber surface tension was used to investigate the water solubility of the characterised bamboo fiber produced by both inadequately treated and getting wet agent treated epoxy. Improvements in the composite interface were verified by measurements of the vibrant contact area and adhesive shear forces. Finally, the tensile properties of composites made from bamboo fiber reinforced epoxy resin (BF/EP) were evaluated after they were prepared using vacuum-assisted resin transfer moulding. Results demonstrated that the connectivity interoperability of BF/EP composites could be greatly enhanced by the accumulation of the co-surfactant. This treatment increased interlayer shear stress of bamboo fiber reinforced epoxy resin composites by 24.36 MPa, the tensile strength by 111.54 Mega Pascal, and the Elastic constant by 66.7% compared to non-treated BF/EP composites [35, 36].

The VARTM method was effective in producing the wettability agent-revised BF/EP composite. The mechanism by which wetting agents enhance the included adhesion effectiveness of bamboo and epoxy fiber composite materials was determined. FTIR and DSC analysis demonstrated that the wettability factor is not contributing to epoxy resin's reaction, despite lowering the epoxy's surface energy, viscous, and density. When epoxy was applied to bamboo fiber after being treated with a 1% wetting agent, the contact angle was stabilised at around 20°, which bodes well for resin saturation. The included shear testing results demonstrated that connectivity among bamboo fiber and epoxy matrix was greatly enhanced after the addition of the wetting element. In the bamboo and epoxy fiber composite system, improvement would result from the natural movement of the wetting agent. As a result of using a wetting agent, the tensile strength, interfacial shear stress, and Young's modulus of the BF/EP composite prepared via the VARTM process were all increased by 165.7, 99, and 67%, respectively, when related to the incurable BF/EP composites. The surfactant's potential to enhance interfacial bonding in structural materials is very high [36].

Bamboo fibers are increasingly being used as structural support in the polymeric matrix because of their outstanding structural characteristics, sustainability, and recycle-ability compared to other natural fiber reinforced polymer composites. The formation mechanism of the bonds between the polymeric matrix, the bamboo fiber reinforcement, and the interface adhesion is a key element in deciding the ultimate composite material's characteristics and performance. Deterioration of composite materials occurs when structure and reinforcement phases fail to adhere to one another, as a result of factors such as moisture content and lignin. Improving interfacial bonding/adhesion is necessary for bamboo-reinforced composite materials to reach their full potential in terms of their properties. This investigation provides a summary of the chemical and interfacial agents used to improve suspense, rigidity, flexural modulus, interfacial shear strength, and so on [37, 38], including mercerization, compatibilizer use, and silane treatment.

2.3 Coir Fiber Composites

Natural fibers are a natural companion to an epoxy resin, making for composites that are both environmentally benign and mechanically superior. Coir fibers' interfacial bond with the resin was improved through the application of microwave healing, chemical methods, acetic alteration, 3-aminopropyltriethoxysilane modification, and an acceptable combination approach. The coir fibers were subjected to single-fiber pull-out tests, and the results of these tests, as well as the results of analyses of the effects of the different therapies on the fibers' characteristics, could be evaluated utilising scanning electron microscopy, Fourier transform-infrared, and X-ray diffraction (XRD). As calculated here, combining alkali treatment with 3-aminopropyltriethoxysilane surface modification increases the adsorbed compressive and pull-out power of 6.728 MPa and 40.237 N mm among coir fibers and epoxy resin. The results of the primary analysis show that the interfacial adhesion can be enhanced by the formation of a greater amount of electronic and biochemical interactions at the interface utilizing this method. This work not only improves our understanding of coir-fiber-reinforced adhesive resin composites but also has significance for the investigation of natural fiber composites [39, 40].

The properties of the coir fiber composites were analyzed and performed extraction tests on separate fibers to examine the impact of eight various operations on the surface adhesive capabilities of coconut fiber in an epoxy resin matrix. All these methods were found to enhance the adhesion properties of the coir fibers with the epoxy resin matrix, as evidenced by single-fiber pull-out tests conducted on several different pull-out specimens. After evaluating A-S-CFPS against similar materials, it was discovered that it possesses exceptional interfacial bonding properties. Compared to U-CFPS, the interfacial shear strength is 6.728 MPa and the pull-out energy is 40.237 N-mm, all significant improvements. After being extracted and examined under a microscope, the fibers and matrix revealed that the coir fiber in A-S-CFPS had enhanced the interfacial characteristics by eliminating the non-cellulose agents and creating adhesive glue with the matrix. Chemical analysis of extracted fibers revealed that 3-aminopropyltriethoxysilane from A-S-CFPS had fortified the ties between the fibers and the matrix. Therefore, alkali treatment and 3-aminopropyltriethoxysilane interface bioactivity can improve the interfacial bonding capacity of coir fibers and the epoxy resin matrix [39, 41].

2.4 Aramid Fiber Composites

Surface modifications with carbon nanotubes, dopamine, and the silane bonding titrant KH560 improved both the synthetic and structural properties of the aramid fibers, as well as the adhesive nature at the interface. Methods such as FTIR, XPS, scanning electron microscopy, and atomic force microscopy were used to examine the fiber's exterior. We measured the modified single-fiber and glass fiber strand specific resilience, surface roughness, hydrophilicity, and interlaminar strength. The modified fiber textile composite laminate was then subjected to uniaxial tensile testing. Experiments show that the surface roughness and resin impregnation capacity of aramid fibers are further enhanced after an initial alteration process involving grafting with silane coupling reagent. Increases of 21.62% in single-fiber specific strength, 73.57% in interfacial shear strength as measured by fiber pull-out tests, and 51.60% in laminate tensile strength are all achieved. This study demonstrates the efficacy of a generic surface modification strategy for a wide variety of fibers, both synthetic and natural. The results presented here contribute to the expansion of a family of high-performance fabric composites that can be used to meet future hurdles in construction management [42, 43].

2.5 Sugar Palm Fiber Composites

For first, second, third or fourth weeks, palm fibers were submerged in salt water before being used as reinforcement in a green bio composite. Natural cassava was used as the composite substance, rather than the usual resin matrix. Scanning Electron Microscopy revealed significant changes in the surface morphology of the fibers and the interfacial bonds of the fiber and the matrix as a result of the immersion treatments (SEM). Longer submersion in salt water improved the quality of the bonds between the surface and the water. The absence of gaps between fiber and matrix indicates that the matrix with 4-week fully submerged fibers has the greatest interconnected edges of fibers and matrix. During the submersion period, the interlocking surface morphology of the fibers could be observed.

As seen in Fig. 4, the interfacial bonds between the sago starch matrix and the sugar palm fiber are quite strong. Due to the high surface tension between the matrix and the fibers that have not been treated, the matrix will not fuse with the fibers because of the pectin film that has been overlaid on their surfaces. Alternatively, as the slits closed, the surface tension was gradually reduced and the matrix-fiber bonding was enhanced through immersion treatments over the following one, two and three weeks. After four weeks of complete submersion, a strong connection is formed, manifested by the disappearance of gaps and the closing of previously visible interfaces. As a result, polymerization reactions between the two substances are stimulated, and the hydrophobic properties of the fibers are altered. Positively charged ions from the fibers and negatively charged ions from NaCl enhance the attachment where the matrix completely enfolds the fibers. Simultaneously, the mechanical interlocking was enhanced by the lowering of lignin and glucans [44, 45].

Sea water was found to be effective at removing unnecessary elements on the fiber surfaces, such as chitin, petroleum jelly, and impurities, and at the same time growing its versatility to connect with the tapioca chemical matrix. An effective morphology may develop over the course of 4 weeks, as evidenced by a growth in superficial coarseness and small pores. Treatment with sea water alters the fiber's surface morphology, increasing the mechanical adhesion impact of the sugarcane bagasse mixture. This innovative study used bio-based matrices, making the bio composite material almost entirely composed of natural substances, which increases the composite's biodegradability and environmental friendliness [46].

2.6 Basalt Fiber

Utilizing Basalt fiber (BF) as a green reinforcing material and plasma polymerization to strengthen the interfacial bonding between BF and polyamide allowed for the development of strong, lightweight BF/PA6, 6 thermoplastic composites. The surface of the BF was modified with 3-aminopropyltriethoxysilane (APTES). Surface BF was subjected to APTES plasma polymerization for 3, 5, 7, and 9 min. This study

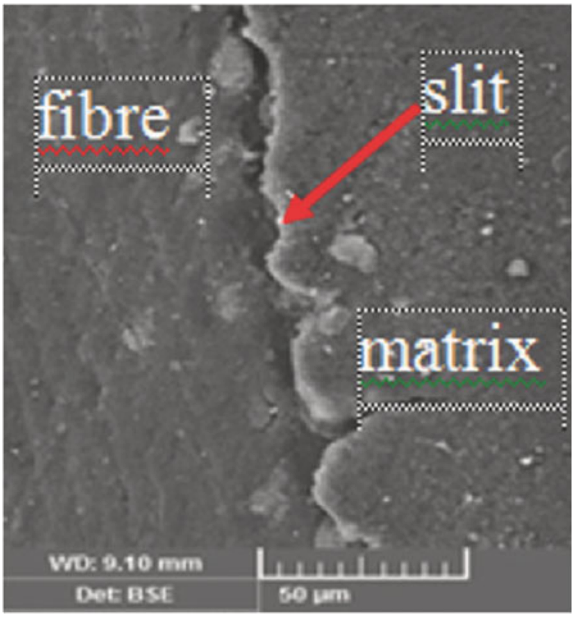

Fig. 4 SEM image of matrix-sugar palm fiber interface [46]

compared the reaction mechanism observed during conventional solution dipping with that observed during plasma polymerization. Investigated the effects of surface chemical modifications on the interfacial adhesion resilience and mechanical characteristics of BF/PA6, 6 composites. Interfacial shear strength was increased by 50.3% and tensile strength by 32.5% when APTES plasma-polymerized BF was compared to untreated BF. Additionally, APTES plasma-polymerized BF performed exceptionally well, outperforming solution-dipped BF in both interfacial shear strength (by 25.7%) and tensile strength (by 15.1%). Since plasma polymerization deposited a creamy synthetic layer on the BF surface that was fully consistent with PA6, 6 and gave the PA6, 6 matrix outstanding physical and toxin bonding, this was the case. Figure 5 shows the SEM fractographs of various basalt fiber composites.

It was found that plasma polymerizing APTES onto the surface of BF increased its interfacial bonding strength with PA6, 6. The composites' interfacial bonding

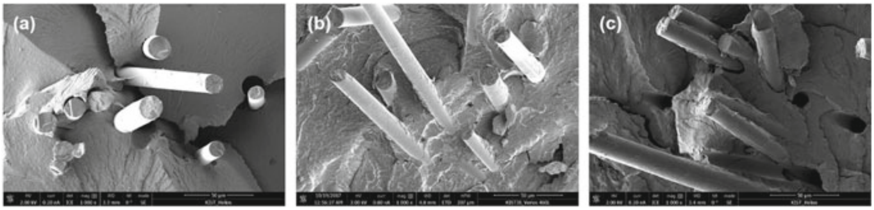

Fig. 5 SEM image of basalt fiber **a** untreated BF/PA6, 6 **b** APTES-functionalized BF/PA6, 6, **c** APTES plasma-polymerized BF/PA6, 6 [47]

strength displayed distinct chemical properties in comparison to that of conventional APTES-functionalized BF, which is also produced via a condensed water response among BF and APTES. A thick polymeric layer, highly compatible with PA6, 6, was formed on the BF surface after 7 min of plasma polymers, because of APTES, which also provided PA6, 6 with outstanding physical and chemical bonding. The tensile strength and IFSS results corroborated these findings, coming in at 50.3% and 32.5% increases, respectively, over the untreated BF. Also, the interfacial shear strength of the APTES plasma-polymerized BF was found to be 25.7% higher and the tensile strength was found to be 15.1% higher than in the solution-dipped sample. These improvements can be attributed to the excellent interphase adhesion that was achieved during the plasma polymerization process. The findings of this study shed light on plasma polymerization as a promising surface treatment for enhancing the interfacial adhesion resilience of BF and PA6, 6 [47–49].

2.7 Root Fiber

Biocomposites made of poly (lactic acid) (PLA) and natural fiber (NF) are biodegradable and sustainably sourced, but they have poor interfacial adhesion. In this work, we used a two-step hydrothermal approach to functionalize ZnO nanotubes on the NF surface, an interaction that was largely driven by the strength of the intramolecular bond. The corresponding composite showed remarkable interfacial adhesion thanks to the seamless inclusion of the root-like NF and PLA composite via the sidewards-grown ZnO nanowires. Significant increases in interfacial shear strength (157%) and debonding energy (403%) especially in comparison to PLA/pristine NF composite provide evidence of this. After performing a single fiber pull-out test and using the finite element method to analyze the fracture morphology, a clear picture of the interfacial reinforcement brought on by the lateral growth of ZnO nanowires was painted. This research can provide a topological architecture interfacial manipulation strategy for enhancing the interfacial interactions of the eco-friendly PLA/NF bio composite, thereby facilitating its widespread use [50, 51]. Figure 6 shows the stress-strain graph of root fiber composites with ZnO nanowire incorporated.

Without any prior treatment, hydrothermal growth generated root-like lateral ZnO nanowires in NFs. Instead of the usual intermediate delamination seen in ordinary NF-reinforced composites, this composite underwent a novel interfacial fracture mode, with breakage of the progressively developed nanowires as the ultimate failure mode. Therefore, the enhanced interfacial characteristic of the PLA/root-like NF composite is not due to communication between PLA and NF, but rather to ZnO nanowires at the interface. Compared to the PLA/pristine NF combination, the shear strength and debonding power were increased by 157% and 403%, respectively, when the amount of ZnO nanowires was increased as depicted in Fig. 7. This study of the weak interfacial interactions between PLA and NF was essential to the efficient production and widespread use of this green polymer combination [51–53].

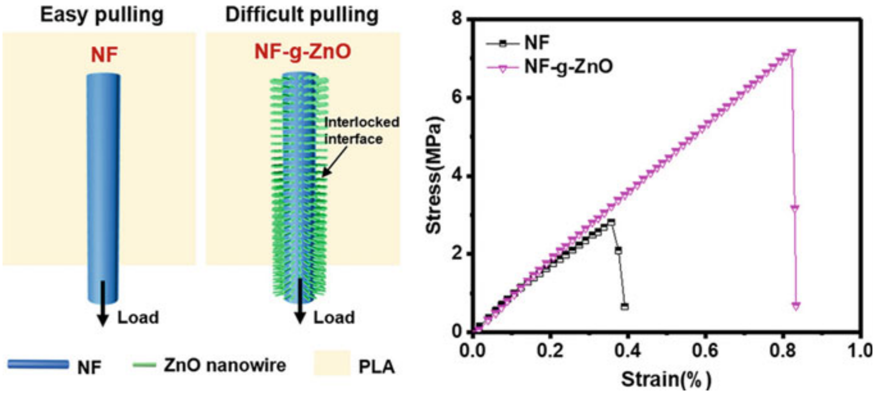

Fig. 6 Root-like NF with ZnO mechanism [51]

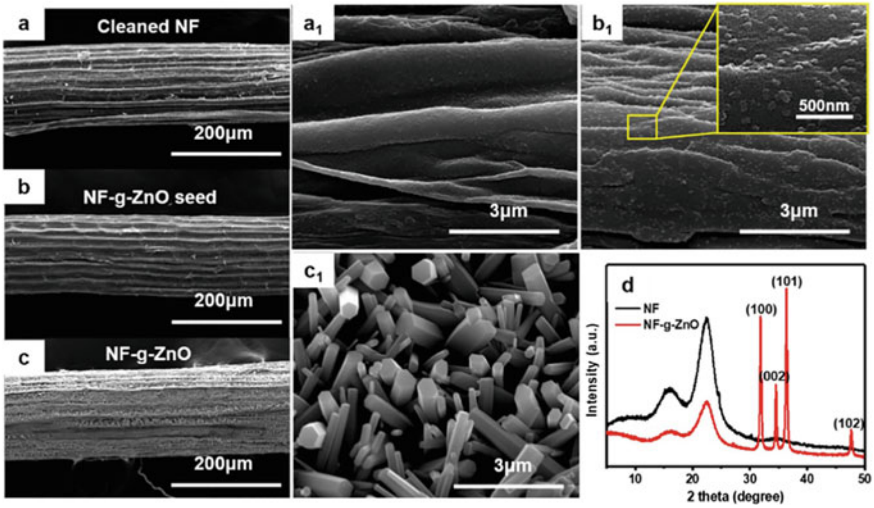

Fig. 7 SEM images of pristine NF (**a** and **a1**), NF with ZnO seed layer (**b** and **b1**) and NF with ZnO nanowires (**c** and **c1**); XRD curves of pristine NF and NF with ZnO nanowires (**d**) [51]

3 Interfacial Bonding of Hybrid Composites

3.1 Flax/Wool Twine Hybrid Composites

Natural fiber-reinforced concrete mixture composites are investigated in this study by introducing 20–30 mm layers of untreated and resin-coated flax/wool twine into various cementitious matrices and then testing the interlayer adhesion between the layers. Crushed fly ash (FA), ground granulated blast furnace slag (GGBS), and ordinary Portland cement were used to create cementitious matrices, and their strength was tested with and without the inclusion of flax/wool twine (1% volume ratio). At 7 and 28 days, these samples' mechanical properties were evaluated. The results showed that the bending and shear strength of untreated NFR samples was lower than that of their unreinforced (UNR) counterparts because of the insufficient adhesion among the untreated fibers and the concrete mixture glue, which led to the formation of voids. The flax/wool twine was then coated with epoxy (EP) and polyurethane (PU) resin before being incorporated into a few cementitious pastes [54].

Bending and yield stress were found to be higher in the 7-day and 28-day tested samples compared to the UNR etc. and treated NFR controls, respectively. At 28 days, the mixture of 50% OPC and 50% FA matrix with epoxy wrapped flax/wool twine achieved the greatest increase in bending resilience, 61% compared to UNR. With the same concrete mixture ratio, flexural strength in PU-coated samples increased by 31% after 28 days. This improved mechanical performance can be traced back to the resin coated NFR samples' morphology, which revealed an intimate interlayer connection to the vicinity concrete mixture dough. Mechanical, physical, and interfacial adhesion characteristics of uncoated and resin-coated flax/wool twine reinforced with different cementitious matrices (OPC, FA, and GGBS) were studied. Mechanical characteristics of cement materials were discussed, along with the impact of various parameters and the potential interactions of resin treated reinforcement [54, 55] (Figs. 8, 9 and 10).

The results of the experimental study indicated some of the following points. The mortar's compressive and flexural strengths may be lower in reinforced samples than in unreinforced controls because of the presence of flaws caused by the uncoated flax/

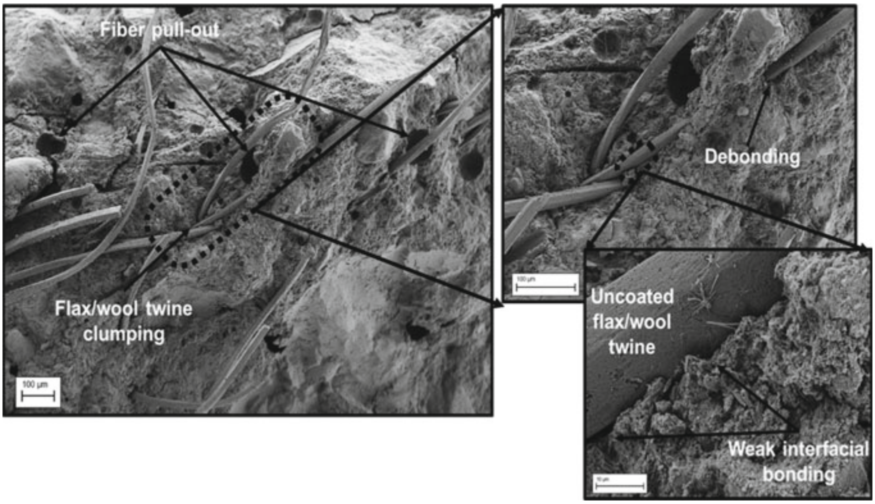

Fig. 8 SEM image of flax/wood twine [54]

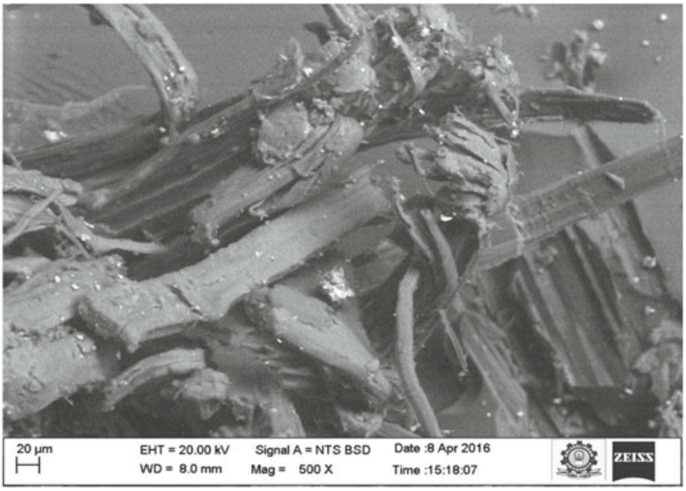

Fig. 9 SEM image of jute/kenaf fiber [61]

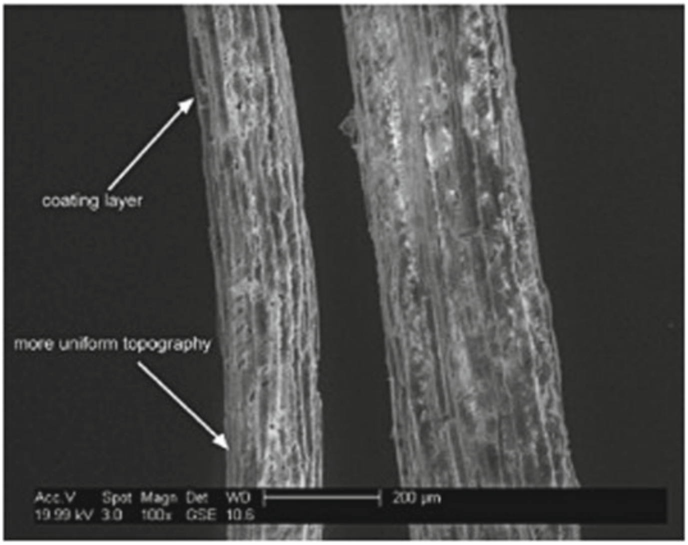

Fig. 10 SEM image of agave tequilana bagasse fibers [65]

wool twine. This is likely due to the untwining of flax and wool twine into individual fibers that occurred during the mixing process, which led to their uneven distribution. Nothing about the observed phenomenon allows for the desired reinforcement effect to materialize. The mechanical characteristics of concrete samples coated with flax or wool resin was better than that of not-reinforced and untreated specimens. Epoxy resin, as a coating material. A cementitious matrix made of OPC and GGBS with a volume proportion of 50:50 performed better than a cementitious matrix made of OPC and FA.

These two matrices have the potential as hosts for cementitious composites reinforced with natural fibers. At the end of the curing time period of 7 days, the NFR cementitious composites with GGBS added to them had a higher level of permeability. The addition of fly ash to the mixtures improved their permeability after 28 days. Microstructural analysis of treated flax/wool twine concrete specimens versus untreated specimens was able to explain the underlying mechanical advancements. More in-depth studies are needed to determine the impact of natural fibers on fracture reduction, concrete structure porous structure, liquid and hypochlorite penetrability, and the future potential of natural fibers in cement materials. The water-holding capacity of the natural fibers can be used in a novel way by employing internal curing technology to create high-performance cementitious composites [56–58].

3.2 *Jute/Kenaf Fiber*

The ability to sustain applied load to the weight and recyclability of natural fiber composite laminates have led to their widespread use in constructional requirements such as aircrafts, automotive, and sports items. Since these composite laminates will be used for such specialised purposes, research into their mechanical behaviours is essential. The objective of this investigation is to characterise the dynamic qualities shared by Jute and Kenaf in their hybrid form. Natural fiber has the major drawback of being Resin incompatible. Fibers can have their surfaces treated to increase their adhesion to resin and decrease their susceptibility to moisture absorption. Hand lay-up is the fabrication method used to create laminates. Several mechanical tests were conducted on jute/kenaf hybrid laminates, including a tensile test, a flexural test, and an impact test. All specimens were prepared and tested for mechanical properties in accordance with ASTM specifications. A scanning electron microscope is used to examine the microscopic structure of the various hybrid specimen layers [59, 60].

The scanning electron microscope was used to analyse the morphology. After testing the composites, the surface characteristics were investigated using SEM. After that, SEM was used to examine the samples more closely. Scanning electron micrographs reveal the strong adhesion at the matrix-fiber interface. Despite careful production, the SEM microstructure of the Jute/Kenaf fiber composite reveals intra fiber delamination, most prominent in the hemp fibers, reducing the composite's strength. Image Description: Hemp fibers are shown running vertically in the upper half, and horizontally in the middle half. In a tensile test, the fibers are typically damaged more along the direction of loading. Impact strength at the hemp fiber fracture point in the KHHK composite. Rapid impact causes fiber fractures, but no signs of fatigue failure can be seen. The fiber's woven structure also makes its matrix distribution and interfacial adhesion obvious. Jute/Kenaf fiber composites are tested for tensile strength, and this investigation examines the fiber-resin adhesion in these materials.

Although some imperfections such as air bubbles and fiber draw-out are present, the adhesion is generally satisfactory. Resin has a smooth appearance, while fiber has a more textured appearance. Hemp's extreme durability comes from the fact that its fibers are so tough that they can withstand being broken apart one at a time without losing their integrity. These high stress values provide further evidence that the fiber and matrix effectively transfer stress in the tensile direction. Image from a scanning electron microscope of a Jute/Kenaf fiber that was broken in a flexing motion. Whenever a load is applied across a section, interphase delamination occurs. Due to the consistent pressure applied to the specimen, the number of voids is discovered to be relatively small. In this case, the crack does not initiate in the glass fiber but rather in the natural fibers, which ultimately fails. Similarly low flexural strengths point to a lack of pressure transmission from the composite to the fiber. Tensile test on a composite of jute and kenaf fibers. If the fibers are unbroken, then the epoxy matrix must be adequately wetting the fiber. Good toughening relies on sufficient interfacial adhesion. Lacking this, the calculated values is related solely to the robustness of the medium on a smaller area due to appearance of the non-supported fibers, which is why fiber peeling and breakage make up such a small fraction of the total failure mode. As a result of the strong adhesion, both the matrix and the fiber can contribute to the structure [60, 61].

The scientific community is struggling to find solutions to the pressing problem of treating solid wastes, especially those made from materials that cannot be broken down by natural means. The costs associated with decomposing these wastes are too high, and the chemicals they generate are harmful. Above ground, the best option is to use natural fibers to reinforce polymers. An entirely new category of composites, based on epoxy and featuring Kenaf and Jute fibers that have been treated, has been successfully fabricated. The mechanical properties of epoxy composites with treated Kenaf/Jute reinforcement are improved over those of untreated composites. This study looked at the impact of treatment on the mechanical characteristics, moisture absorption property, and morphological nature of resin strengthen with original fibers. Fibers with higher percentage celluloses were produced after surface treatments removed starch, glucans, and other non-cellulosic substances.

Chemically treated fibers had a rougher surface and a larger effective surface area, which improved their interaction with the matrix. Results indicated that the mechanical strength of the Kenaf/Jute composite was the highest. The morphological evidence supported the mechanical findings. These studies show that treated fibers when combined with the right amount of coupling agents and fibers, can strengthen an epoxy matrix. Because of their superior mechanical properties compared to those of conventional fiber-reinforced polymeric materials, hybrid fiber-reinforced epoxy composites may find wider use in automobiles and constructing product lines industry sectors [62, 63].

3.3 Agave Tequilana Bagasse-Fibers

The possibility of making environmentally beneficial composites out of tequila production waste fibers. In this study, we examined the potential of four different naturally sourced fibers with surface changes for application in the production of eco-friendly composites. Before receiving any kind of surface treatment, the morphology and tensile qualities of the agave bagasse fibers used in tequila production batches were examined using optical and environmental scanning electron microscopy (ESEM) and a single fiber tensile test. Four alternative surface modifications, including alkali, acetylation, enzymatic, and silane, were applied to agave fibers to improve their surface properties and compatibility with polylactic acid (PLA). We analysed how the fibers' morphology, mechanical properties (i.e., Youngs modulus and maximum tensile properties), interface resilience (IFS), and moisture content changed as a result of these treatments [64, 65].

Agave bagasse fibers' tensile strength, modulus, and IFSS were all improved by surface treatments, with the greatest findings coming from alkali-treated fibers (final ultimate tensile of 119.10 MPa, Young modulus of 3.05 GPa, and IFS of up to 60% higher, 5.21 MPa, than untreated samples). Results from these tests indicated that an alkaline treatment is the most effective for agave bagasse fibers. These findings illuminate the potential for recycling these agave fibers as part of the production process of PLA-based green composites by illuminating the adsorption bonding among agave bagasse fibers and PLA.

ATF (Agave tequilana fiber), a byproduct of the tequila industry, can be used as a reinforcement fiber in PLA-based green composites. Because of its minimal price, reduced intensity, high hardness, reasonable specific intensity characteristics, and biodegradability, PLA can be used in a variety of green composite applications. For future ATF-based green composites to perform at their best, the adhesion between the PLA matrix and ATF is essential. There are treatments that can be applied to the surface to help with this. Surface modification methods enhance the tensile strength of ATF, the bonding strength between PLA and ATF, and the absorption properties of ATF. In comparison to the UNF samples, AKF showed superior results for maximum stress and shear strength by roughly 50 and 60%, in both. Therefore, using treated ATF could result in a higher fiber load content. It is believed that the major elements trying to improve interfacial bonding are the enhanced coarseness in the exterior region of the fiber and the withdrawal of the non-cellulosic materials evidenced by alkali and enzyme-treated fibers. ACF performed the best in terms of water absorption, with only an 18% decrease compared to untreated fibers. As the fiber degrades from water absorption, the mechanical properties and the use of ATF in fiber-reinforced composites are jeopardized. In the case of biodegradable composites, however, this may prove useful. In conclusion, a green composite material made from treated ATF and PLA is possible to imagine, with the added benefit of being able to perform well under low-stress circumstances in the absence of jeopardizing the ultimate composite goods or the surroundings [66, 67].

4 Conclusion

This chapter deals with the discussion of various specifications of fiber and matrix bonding and the analysis of the interfacial bonding using SEM images. Abaca fibers' exterior characteristics and achievement were found to be greatly enhanced after being treated with low concentrations of alkali which enhances the interfacial bonding. Bamboo fiber provides a summary of the chemical and interfacial agents used to improve uncertainty, rigidity, flexural modulus, and interfacial shear strength including mercerization, compatibilizer use, and silane treatment. Interface bioactivity can improve the interfacial bonding capacity of coir fibers and the epoxy resin matrix treated with alkali and 3-aminopropyltriethoxysilane. The results presented for Aramid fiber, contribute to the expansion of a family of high-performance fabric composites that can be used to meet future hurdles in construction management. Sugar Palm Fiber, used bio-based matrices, making the biocomposite material almost entirely composed of natural substances, which increases the composite's biodegradability and environmental friendliness. Flax/wool twine and water-holding capacity can be used in a novel way by employing internal curing technology to create high-performance cementitious composites. Because of the superior mechanical properties of Jute/Kenaf fiber, compared to those of conventional fiber-reinforced polymeric materials, hybrid fiber-reinforced epoxy composites may find wider use in the automobiles and constructing product lines industry sectors. Agave tequilana bagasse-fibers, a green composite material made from treated ATF and PLA is possible to imagine, with the added benefit of being able to perform well under low-stress circumstances in the absence of jeopardizing the ultimate composite goods or the surroundings. The findings of Basalt fiber, hut light on plasma polymerization as a promising surface treatment for enhancing the interfacial adhesion resilience of BF and PA6, 6. The weak interfacial interactions between PLA and NF were essential to the efficient production and widespread use of this green polymer combination.

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New Methodologies to Improve the Interfacial Interaction in Natural Fibre Polymer Composites

Mahmut Ali Ermeydan

Abstract In recent years, because of rising environmental consciousness, depletion and emission issues of oil resources arose several research and production attempts to use natural fibres instead of synthetic fibres in fibre reinforced composites. Natural fibres can be a sustainable resource for composites with several advantages such as being biodegradable, cheap and available, having acceptable strength to weight ratio, low density, excellent insulating/noise absorption and reduced tool wear. Researchers increasingly explore and try to find novel and appropriate methods to utilize natural fibres in various industrial applications. However, plant based fibres have some drawbacks due to especially its chemical structure. When compared to its synthetic competitors like glass fibres, natural fibres have poor moisture/water resistance that causes swelling, poor fibre/matrix adhesion, low impact resistance, low durability, restricted maximum processing temperature, poor fire resistance, and quality variability. Among those disadvantages, it is fundamental to resolve problems of fibre-matrix interactions to produce a qualitative composite that meets certain specifications. Several researches revealed that improving the interfacial interactions in natural fibre-matrix composites by establishing better stress transfer results better physical, mechanical and thermal performance. Therefore, modifying the natural fibre surface through several methods in order to improve adhesion of fibre and matrix for successful utilization of these materials in different applications. This chapter provides a review of existing research that points out surface modification or treatment methods of natural fibres and underlines recent methodologies established improving interfacial interactions between natural fibres and matric for composite applications.

Keywords Interfacial interaction · Adhesion · Surface modification · Fibre-matrix interface · Natural fibre composites

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1 Introduction

Polymer composites are used in almost all material fields (home appliances, packaging, car, aviation, transportation, sports, entertainment, etc.). Almost all synthetic and biobased polymers (elastomers, thermosets and thermoplastics) can be used as matrix material to cover the reinforcement or filler materials [1].

Beside from inorganic powders [2], or man-made fibres such as glass fibre, silicon carbide fibre, or organic-based fibres such as aramid or carbon fibres have been widely utilized in the form of individual fibres, as mats or fabrics. Among other natural fibres (silk or wool), cellulosic fibres have a hierarchical structure with embedded cellulose fibrils into hemicelluloses and lignin matrix [3]. Cellulosic fibres can be obtained from the stem (hemp, flax, jute, etc.), leaf (henequen, sisal, etc.), seed (cotton, kapok, etc.) or fruit (coir) of a plant [4]. The chemical composition of cellulose fibres vary between different plants and even at different regions of an identical plant [5]. Except from cotton, the chemical content of natural fibres are composed of cellulose, hemicellulose, and lignin as main components, and fibres have little amount of wax, pectin or some extractive materials [6]. Common natural fibres with compositions are listed in Table 1.

In addition to its excellent material properties, sustainability, renewability, and availability around the world are depicts ecological and economical value of the cellulosic fibres [10]. However, due to their hygroscopic and hydrophilic chemical structure, wood and cellulosic fibres have low dimensional stability and need protection to overcome weathering factors (water from rains, erosion from wind, UV light from sun, etc.) [9, 11]. Several protection methodologies have been proposed to manage intrinsic drawbacks of natural fibres, which will be discussed in following section of this chapter. To get an advantage of natural fibres in composites, the problematic features of natural fibres should be resolved. Wood and cellulosic fibres have been used in the industrial level as chips (originally formulated in late nineteenth century Germany) in combination of thermoset resins such as urea-formaldehyde or phenol-formaldehyde, or several other resins [12]. Today, chip and particleboards are extensively used for furniture, interior wall and ceiling lining, countertop substrate, floor decking, or roof sheathing by utilization of wood or agricultural wastes [13–15]. By increasing production and use of thermoplastic boards, the idea of making them cheaper and stable in all manner have been led to the wood plastic composites. The ability of wood composites to be tailored to specific uses with their mechanical strength and price makes them an industrial scale alternative to solid wood, timber or plastic lumber [16–18]. Research efforts carried out over the past 40 years have emphasized mainly compost of natural fibres in polyolefins due to ease of production by extrusion or injection methods and tailoring ability of the composite with various properties (shape, color, etc.).

Natural fibres with excellent mechanical properties has been measured in several researches. For instance, the tensile modulus of flax has been reported comparably close to glass or kevlar fibres in many articles [19, 20]. In terms of a composite reinforcement, it is more realistic to compare natural fibres with glass fibre due to its wide

Table 1 Common natural fibres with compositions and type of the source [7–9]

Fibres		Cellulose (%)	Lignin (%)	Hemicellulose (%)	Pectin (%)	Waxes (%)	Ash (%)	Silica (%)
Bast fibres	Flax	70	2	18	2	1.5	12	–
	Kenaf	45	15	22	6	0.5	4	–
	Jute	60	16	18	8	0.5	4	–
	Hemp	70	7	18	2	0.5	4	–
	Ramie	80	1	14	2	0.3	5	–
Leaf fibres	Abaca	60	10	16	0.5	0.1	2	–
	Sisal	65	11	18	5	1	2	–
	Henequen	75	13	6	–	–	–	–
	Banana	55	18	13	3	4	2	–
	Pineapple	68	7	80	1	3	2.5	–
Stalk fibres	Rice (stalk)	35	14	26	–	–	18	12
	Wheat (stalk)	45	18	29	–	–	6	5
	Oat (stalk)	40	18	34	–	–	7	5
	Bamboo	55	20	35	0.5	–	4	0.5
	Sugarcane	40	22	30	–	–	3	2
Seed fibres	Cotton	92	2	5	0	0.6	1	–
	Kapok	64	23	13	23	–	–	–
Fruit fibres	Oil palm	55	19	25	–	0.6	3	–
	Coir	30	41	14	4.7–7	–	–	–
Wood fibres	Hardwood	40	25	23	0.5	–	0.5	–
	Softwood	42	32	10	0.5	–	0.5	–

*Component contents are expressed as average values from the reported articles

utilization and price in the automotive and plastic industries [21]. Composites reinforced with natural fibres have lower density but similar mechanical performances as glass fibre reinforcement. Another advantage of natural fibres is lower abrasion of processing equipment. Additionally, the composite produced from polyolefins and natural fibres may be recycled or incinerated with releasing any toxic residues. Today, the rise of the biopolymers also create an opportunity to directly compost of green composites into soil to lead complete biodegradation of the composite [22]. Another issue that makes natural fibres a serious alternative to glass fibres is their environmental superiority in terms of used energy during production or emissions [23]. The advantages of natural fibres against glass fibre mainly match the needs of automotive industry, which requires recycling, good specific strength, being lightweight and non-toxic [24–26]. Bhattacharjee and Bajwa reported successfully that recycling of wood-HDPE composite is quite possible by several cycles with about 10% loss tensile strength after 6 cycles [27].

The performance of natural fibre composites is assessed by combination of several factors. Features of matrix polymer and reinforcing fibres, but predominantly the interfacial interaction of the fibre and matrix (e.g., mechanical interlocking, chemical interaction and adhesion) are some of the key factors of composite success [28, 29]. Hundreds of reports have confirmed that natural fibres have great potential as a composite reinforcement material if their inherent flaws could be overcame [29]. Lignocellulosic fibres, like flax, hemp, kenaf, jute, henequen, sisal, coir, bamboo, or wood in their natural state, and other lignocellulosic wastes have been used as reinforcement material with thermoplastic and thermoset matrices to serve as many products [30]. Even waste from pulp and paper industry can be used to produce green composites as reported by Zhang et al. [31]. He reported that higher tensile strength and modulus were obtained by using pulp and paper industry waste.

In many occasions, the difficulty that limits broad utilization of natural fibres in composites is the missing adhesion of fibres with most polymeric matrices. The hydrophilic chemical structure of lignocellulosic fibres hinders better adhesion in a low polar or non-polar hydrophobic matrix polymer resulting in poor interfacial interaction, which ends up with low mechanical and physical features for the composite. To overcome the adhesion problem and boost interfacial interaction, researchers have been suggested several methods to modify cellulosic fibre surface [30, 32]. For most of the examples, modification processes not only improve fibre-matrix interactions, but also make cellulosic fibres more hydrophobic by changing surface chemistry [33]. Zaman and Khan reported recently that acetylated banana fibre reinforced composites absorbed less water than alkali-treated and untreated composites [34].

Assurance and optimization of a better interfacial bonding between fibre and polymer matrix for composites, generally provide optimal mechanical performance (strength, wear, impact, etc.) as well as durability of fibre-reinforced composites. The quality of the final product is critical for in real life applications, therefore it is known that most of the wood plastic composite producers (for decking, etc.) need to use compatibilizing and/or coupling agents to maintain long-term warranties up to 35 years [35].

Several publications have been reported on use of coupling agents for compatibilization of natural fibres or fibre surface modification in natural fibre-polymer composites (Fig. 1). There is an obvious increase on research about use of coupling agents or fibre modifications in order to improve adhesion or fibre properties. Bao et al. reported grafted acrylamide on to cotton and jute fibres by horseradish peroxidase (HRP)-initiated polymerization and well-verified polymer existence onto fibre surface by Fourier transform infrared spectrometer (FTIR) and X-ray photoelectron spectroscopy (XPS) [36]. Chanka et al. reported modification of pineapple leaf fibres with aminosilanes as adsorbents for H_2S removal [37]. Liu et al. modified lyocel fibres by phytic acid (PA), which possesses high phosphorus content and has potential flame retardancy [38]. Those examples have been focused on modification of plant fibres for purposes other than composite manufacturing. There are also a wide research interest nowadays on modification of plant fibres in order to use them in polymer composites [39–43].

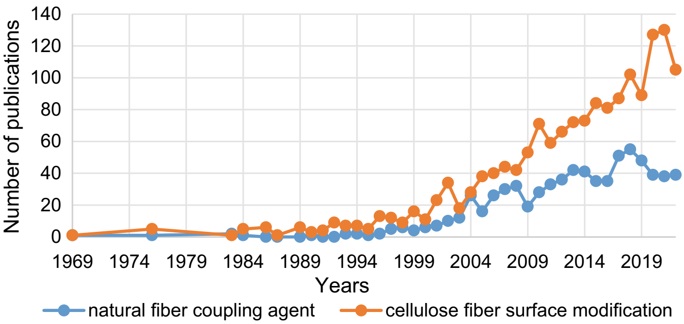


Fig. 1 Numbers of publications related to use of coupling agents and surface modification of cellulosic fibres (scopus search)

Ben Abdallah et al. impregnated flax fibres with various monomers and polymers and cured them with electron beam and radical polymerization (Fig. 2). They revealed that the impregnation system (solvent and monomer type) affects the regioselectivity of the modification. The distribution of the monomers and polymers were analyzed by SEM-EDX to characterize phosphorus molecules.

In the past decades, conventional polymer matrices (polypropylene, polyethylene, polyvinyl chloride, unsaturated polyester, epoxy, etc.) reinforced by modified jute, flax, hemp, wood or other natural fibres for biocomposite production by using both modification techniques or coupling agents have been well documented [44, 45]. One of the initial examples of fibre modification for composite purposes was reported by Zadorecki and Flodin. Dichloro-s-triazine derivatives were reacted with cellulose fibres, further styrene were grafted to form polystyrene onto cellulose surface and

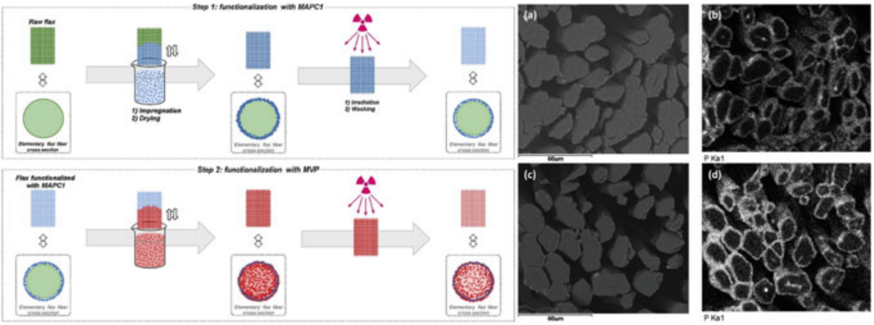


Fig. 2 (Left) schema of multi-step procedure of two different polymer grafting. (Right) **a** and **b** SEM observations and phosphorus mapping using EDX analysis of flax fibres obtained after using MAPC1(Dimethyl (methacryloxy) methyl phosphonate), (**c**) and (**d**) respectively SEM observations and phosphorus imaging using EDX analysis of fibres obtained after using MVP (Dimethylvinyl phosphonate). Picture adapted from Ref. [42] with permission from Elsevier

novel material were characterized by infra-red spectroscopy and XPS. The modified cellulose were proposed to be suitable as a coupling agent in order to enhance adhesion between cellulose and polyester resin [46]. Zadorecki and Floding further tested mechanical features of modified cellulose-polyester composites and tested ageing in order to investigate debonding at the fibre/matrix interface using both optical microscopy and SEM. They found that modification both improved mechanical and weathering resistance of the composites significantly [47, 48]. Today, after initial researches of silane treatment [49], still use of silane coupling agents have room for further investigations. Anaya-Ramirez et al. recently reported static characterization and fatigue behavior of silane treated henequen fibre-reinforced-HDPE composites. The mechanical stress test results showed that the interfacial bonding resistance improved by 30% due to the additional chemical treatment and improved fibre/matrix interaction. In the study, it was shown that NaOH and silane treated henequen-reinforced composites exhibited enhanced resistance under cyclical loads [50].

This chapter will present the basic principles of fibre-matrix interface and adhesion, further will summarize old, continuing and new methodologies of natural fibre treatments for improvement of adhesion in polymer composites (Fig. 3). The nature of the cellulosic fibres should be known in order to assess and figure out the problems of natural fibres in composites. The adverse surface chemistry of natural fibres and matrix polymers needs understanding of interface interactions, thus various methodologies that have improved fibre-matrix adhesion have been investigated. Not only chemical bond formation or grafting methods (anhydrides, silane or vinyl polymer grafting) [42, 51], but also physical methodologies (plasma, corona, etc.) have been proposed so far [52–54]. Recently several aspects of biocomposites produced by novel fibre treatments to improve fibre-matrix adhesion have been also assessed. This chapter has been summarized both from the chemical and mechanical scope to reveal researchers' point of view.

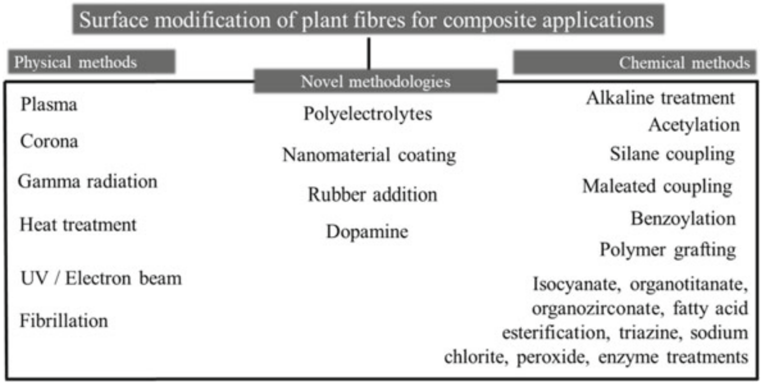


Fig. 3 Various types of modification methodologies for plant fibres surface

2 The Fibre-Matrix Interphase

The critical disadvantages of cellulosic fibres emerge due to polar nature of cellulose, which decrease the dispersibility of cellulose fibres in non-polar polymers. Moreover, hygroscopic nature of plant fibres limits their utilization in outdoor applications. Various polymer types have been used so far with natural fibre reinforcement [10, 33, 55–57]. Polyethylene (PE), polypropylene (PP), polystyrene (PS) and recently polylactic acid (PLA) are common thermoplastic polymers that have been used as matrix for biocomposites [58–60]. As thermoset matrix, the most frequently used resins are epoxy, phenolic and unsaturated polyesters [61]. Having different chemical structure of those polymers results different affinity between the fibre and polymer. It was introduced by Joseph et al. that among phenol-formaldehyde, polyester and epoxy matrices used with sisal fibre, phenolic resin showed better tensile and flexural features due to the high interfacial bonding. Nevertheless compared to thermoset resin composites, mechanical properties of sisal reinforced LDPE improved largely due to the high ductility of LDPE and strength to modulus ratio of sisal fibre [62].

Investigation of several other combinations of natural fibre reinforced thermoplastic or thermoset composites has been continuing. Here are some recent examples. Asim et al. reported the effect of different amounts of NaOH treatment of palm fibre on mechanical and thermal features of phenolic composites [63]. He found that the alkali treatment significantly improved tensile properties at 50% fibre loading. Sarikaya et al. produced natural fibres reinforced epoxy composite and investigated mechanical properties (Fig. 4). He used resin transfer molding technique (RTM) to produce composites reinforced by eucalyptus, birch, palm fibres. The tensile strength of plant fibre reinforced composites were found as 29.5, 42.2, and 45.3 MPa, respectively, where bending strength of composites were found as 58.8, 68.6, and 79.9 MPa, respectively. The impact energy of the birch composite was measured as 0.11 J, while palm and eucalyptus composites were measured as 0.13 and 0.12 J, respectively. It was stated by the authors that type of the fibre was found efficacious in the mechanical properties of composites [64]. The example shown the adaptation of RTM process with natural fibres that is important from an industrial viewpoint.

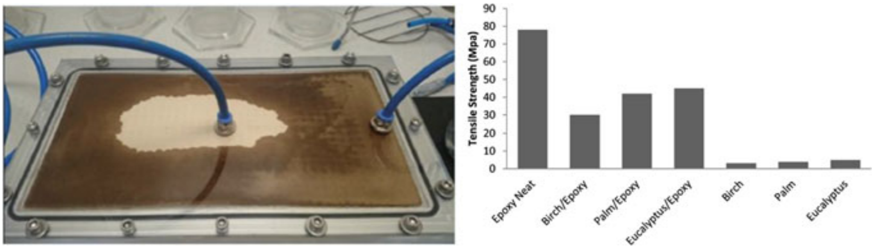


Fig. 4 RTM set-up and tensile strength of birch, palm, and eucalyptus fibre reinforced epoxy composites. Picture adapted from Ref. [64] with permission from Elsevier

Still there is huge room to understand fibre-matrix interface and improvement of adhesion. Basic principles of fibrillation just by alkali treatment of hemp fibres improves mechanical properties. Mazzanti et al. investigated the reinforcing mechanisms PLA biocomposite reinforced by untreated and 3 and 6 wt% alkali-treated hemp fibres. She reported that tensile features of treated hemp/PLA composites were higher than controls. The reason that researchers explained was the opening of fibre bundles and individualization of micro fibres due to alkaline treatment coupled with shearing of extrusion compounding [65].

One of the very basic and clear presentation, which shows improvement of fibre-polymer interaction, has been presented by Liu et al. Corn stalk treated with silane has been used as potential reinforcement material in rubber polymer composites (Fig. 5) [43]. It was reported that the composites produced with the reinforcement of silane treated fibres has 20–97% more tensile strength than control samples, while tensile modulus improved between 70 and 90%. The impact strength of the composites increased by 5–52% compared to controls. Morphological studies revealed that the fracture surfaces of both silane treated fibre composites present better fibre-matrix interfacial bonding compared control samples (Fig. 5, right).

The degree of adhesion represents the extent of stress transfer at the interface between fibre and matrix in composites [21]. Stronger adhesion at the interface implies effective stress transfer and load distribution across the interface. This requires strategy development to modify surface of cellulosic material, thereby achieving efficacious control over the fibre/polymer interface. To improve the physical and mechanical features of composites, a dense or sparse coating of compatibilizers or coupling agents is usually applied that forms or improves chemical interaction between the fibre and the matrix. In the following parts, in the light of the recent researches, which reveal the effects of coupling agents/compatibilizers or coatings on plant fibre reinforced polymer composites and their influence on various properties

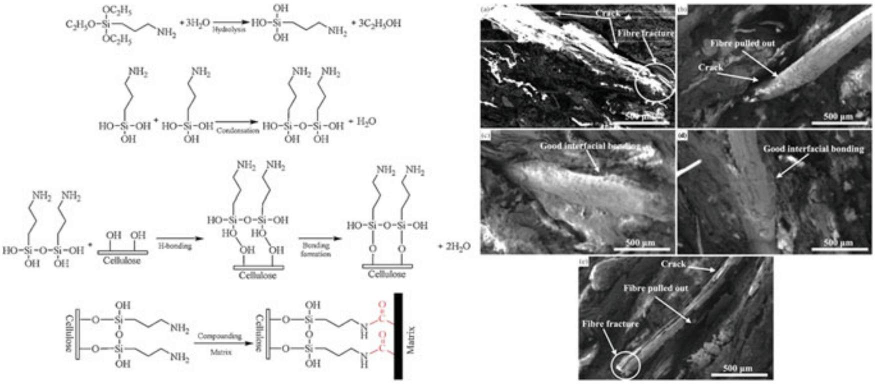


Fig. 5 (Left) Schematic representation of reaction mechanism of silane treatment of cellulosic fibres in polymer composite. (Right) fracture surface morphologies of untreated (**a**) and silane treated (1-5-9-13% silane loading) corn stalk reinforced polymer composites. Picture adapted from Ref. [43] with permission from Elsevier

will be discussed. Even the polymer additives like organotitanates, organozirconates or N-substituted methacrylamide have been studied and their effect on interface interactions of natural fibre-polymer composites was reported [66, 67]. However, there are a few studies of their use in natural fibre composites, thus more prominent and promising methodologies will be discussed here, as well as novel methodologies to improve the interfacial interaction in natural fibre polymer composites.

3 Fibre Surface Modification Methods

The market of natural fiber composites has been growing for a couple of decades in construction, wind turbine, and predominantly automotive markets. Increasing environmental concerns and demand of lightweight materials for energy efficiency increased attention on natural fibres. However, in many occasions hydrophilic and hygroscopic nature of plant fibres limits its utilization and have to be control for better adhesion in polymer or cement matrices. For this purpose, plant fibres can be modified by physical/physicochemical or chemical methods in order to improve surface affinity in polymer/cement matrices (Fig. 3).

3.1 Physical/Physicochemical Methods of Modification

Physical treatments of natural fibres can change structure and surface features of the fibres and therefore may improve especially mechanical bonding of fibres with the matrix. It is known that refining by mechanical means is one of the key steps to produce stronger paper products [68]. Because fibrillation of cellulose increases surface are and during paper formation cellulose fibrils bind together from many points by mainly hydrogen bonding, thus strong paper products can be obtained. Similarly, lignocellulosic plant fibres can also be fibrillated for improved surface area. Senthilkumar et al. reported mechanical refining of the coconut fibres to use them in epoxy matrix. He found that the refining provided distribution of the fibres homogeneously and significantly improved tensile and impact properties of epoxy composites compared to controls [69]. Another study suggests close results. Bernaoui et al. fibrillated surface of flax fibres with a PFI mill and measured tensile properties. The results indicated a raise in tensile properties up to 500 mill revolutions, but more refinement caused reduction of tensile strength [70]. These recent studies are important because may open a door to a newer research topic on mechanical pretreatment of fibres for mainly short fibres in order to make short fibre based bulk, press, extrusion or injection molded composite products. In heat treatment, fibers are heated to a temperature close to fiber degradation. However, there are very limited research on this subject. Heat treatment possibly effects reduction of water uptake of fibres, but reduces strength and cellulose crystallinity [64].

Another physical or more physicochemical process is electric discharge and radiation-based methods (Plasma, Corona, gamma radiation, electron beam, UV, etc.). These treatments changes the surface and structural features of plant fibres without use of any chemicals and consequently improve interactions between fibre and matrix. As noted, there are various types of physiochemical treatments such as corona, plasma, UV, e-beam and gamma radiation to change structure, surface energy or chemical functionalities of plant fibre surfaces [71]. In plasma treatment, an electric field is applied to produce electric current with highly charged ions, electrons or radicals. These energetic particles collide to surface and change functionality of the surface of the natural fiber by means of chemical structure. Pressure level, gas type (O_2, N_2, CO_2, Ar, etc.), flow speed, and power can be controlled [72]. On the other hand, Corona treatment is the simplest way of plasma treatment. Air is used for ionization and created particles colloid the surface with electric discharge. The fibre surface energy is changed by both plasma and corona treatments. There are limited examples of corona treatment so far and successful examples are rare [73]. One of the articles on corona treatment of hemp fibres in order to reinforce polypropylene matrix were reported improved mechanical results for composites [74]. Ragoubi et al. found that the corona treatment of fibres enhanced tensile strength and modulus of composites about 30% compared to controls. On the other hand, more research have been conducted on cold plasma treatment most probably due to better results can be obtained by plasma method [71, 75, 76]. Sinha and Panigrahi reported that flexural strength of plasma treated jute fibre-polyester composite improved about 15% compared to controls [76]. Recently, Gupta and Tiwari reported the cold glow discharge argon plasma modification of sisal fiber enhanced interlaminar shear strength, flexural strength, and tensile strength of the epoxy composites by 50%, 49%, and 31%, respectively [77].

The energetic UV light provokes surface oxidation and increase polarity, which provides better wettability and strength in composites. Gassan and Gutowski reported a successful example of UV exposure of the jute yarn, which obtained up to 200% higher polarity compared to controls. The treatment improved flexural strength of the composite of about 30% at optimum treatment conditions [78]. UV, e-beam as well as gamma radiation are also used to enhance toughness or strength of the composite material by making additional chemical bonding at the interface [79].

3.2 Chemical Modifications

Chemical modification methods applied on natural fibers are generally significantly effective methods than physical modification methods and considerably improve the adhesion of natural fibres and polymeric matrix. To date, comprehensive research have been conducted to reveal the impacts of chemical modification on natural fibers and their composites. The chemical difference of natural fibre and polymer, which are considered as two different phases, ends up with poor interactions at the fiber-matrix interface [80]. The hydrophilic chemistry of the plant fibres can be changed by the chemical modification or treatments and intermolecular interactions between fiber and polymeric matrix can be improved [4, 28, 45, 62, 81]. On the other hand, the very basic treatment by alkaline solution (generally NaOH) works simply and effectively by removing waxy substances/partially lignin and hemicellulose on the surface, thus increasing the surface roughness of the fibre surface, which provides matrix polymer better mechanical interlocking with fibre [80]. Chemical treatment methodologies may function different as observed in alkaline example. However, most of the methods are based on the chemical substitution or addition reactions to establish ester or ether bonds between hydroxyl groups of natural fiber polymers (cellulose, hemicellulose, or lignin) and introduced chemical [30, 40, 71, 79, 82, 83], even the alkaline treatment. The chemical treatments of natural fibres for composites have been varied and specialized by the utilized chemicals and some of them are called acetylation and other anhydrides, silane, benzoylation, maleated coupling agents, acrylic-acrylonitrile or vinyl polymer grafting, isocyanate, peroxide, sodium chlorite, stearic acid, triazine and further polymer grafting, fatty acid chlorides, and fungal treatments, etc. [80, 84–89] (Fig. 6).

Excellent research articles have been published so far on these modification or treatment methods. For instance, Smith et al. reported agave fibre reinforced poly (3-hydroxybutyrate) (PHB) biocomposites. She used organic peroxide as compatibilizing agent for composite and optimized the amount of peroxide as 0.1 phr,

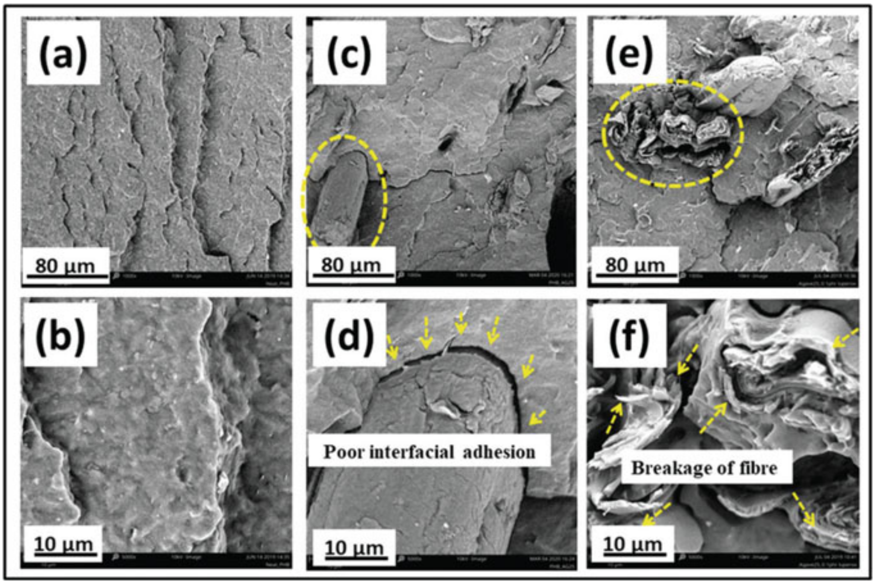


Fig. 6 SEM images of fractured surfaces **a** 1000× and **b** 5000× magnifications of neat poly (3-hydroxybutyrate), **c** 1000× and **d** 5000× magnifications of poly (3-hydroxybutyrate) agave composite 75/25/0 and **e** 1000× and **f** 5000× magnifications of poly (3-hydroxybutyrate) agave composite 75/25/0.1 phr at and, respectively. Picture adapted from Ref. [84] with permission from Royal Society

which significantly improved flexural and impact strength of the biocomposite [84]. Recently, Gieparda et al. reported silane modification with combination fire retardants of flax roving and fabric in order to improve flame retardant efficiency epoxy composite [40]. She stated that flammability of reinforcing fibres was reduced by 400%. However, the pros and cons of these treatments have already been reviewed dozen times [5, 7, 9, 24–26, 30, 33, 61, 68, 71, 90–92], thus new methodologies will be discussed in the following sub-head.

3.3 New Methodologies

The replacement of glass-fibre sheet molding compound products by using natural fibre-reinforced composites in the automotive/electronic/construction industries has been one of the enthusiastic motivation for plant fibre research. High strength to weight ratio, biodegradability, recyclability, sustainability, being CO_2 neutral and worldwide abundance of plant fibres draws attention for many years. Today, it is fact that natural fibres can be used in many applications, but for longer service life and to make advanced composite materials, natural fibres should be somehow modified. The conventional modification and compatibilization methods in composite researcher still study on new methodologies for improved fiber-matrix interactions. In this part, some of new methodologies in order to increase adhesion of biocomposites will be summarized.

3.3.1 Polyelectrolytes

Use of a Layer by Layer deposition of polyelectrolytes on natural fibres has been investigated for a few years. Especially flame retardancy and heat resistance of natural lignocellulosic fibres can be improved, but there are examples that showed several properties of composites can be enhanced by polyelectrolyte modification [93]. Polyethylenimine (PEI) and ammonium polyphosphate (APP) are common polyelectrolytes used with natural fibres. Polyelectrolytes can also be used together with inorganic nanomaterials, which additional features can be added to the fibres.

Lin and Renneckar melt compounded polypropylene and modified fibre (25 wt%) by montmorillonite clay and poly (diallyldimethylammonium). They suggested a novel methodology but could not found any enhancement on properties of composite [94]. Later, Ding et al. reported an anionic ammonium polyphosphate (APP)-cellulose nanocrystal hybrid in order to use it to enhance properties of wood-HDPE composite (WPC) (Fig. 7). Ammonium polyphosphate (APP) and cellulose nanocrystals (CNC) were used to form a hybrid material by self-assembly with ionic and hydrogen bonding interactions. Addition of 20% APP(18%)-CNC(1.5%) hybrid increased the tensile strength and Young's modulus of WPC by 42.7% and 75.9%, respectively. He found that the whole materials have both high load bearing capacity

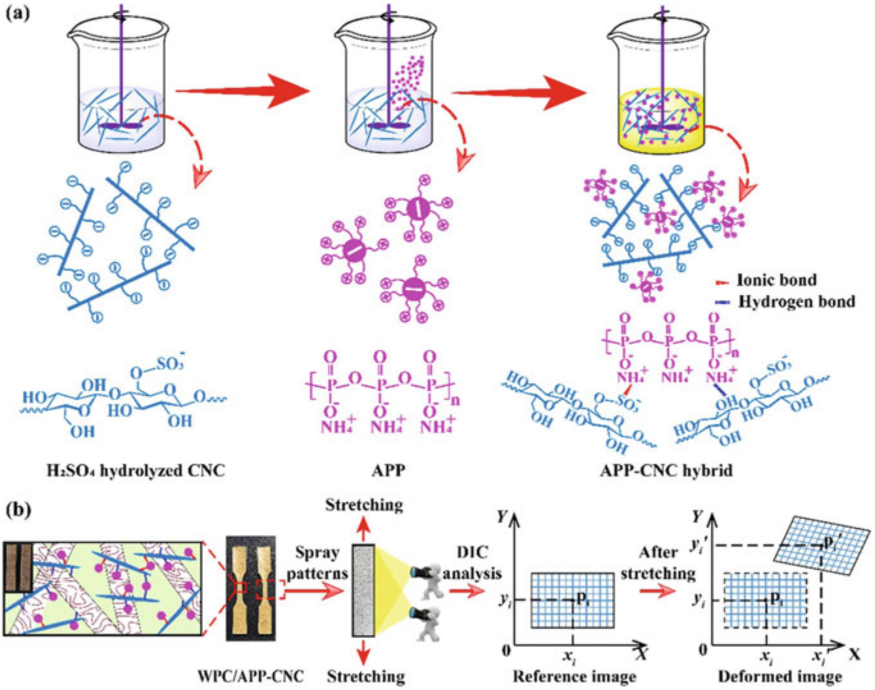


Fig. 7 Schematic representation of **a** an anionic polyelectrolyte hybrid preparation and **b** digital image correlation. Picture adapted from Ref. [94] with permission from Elsevier

and resistance to deformation. He also used digital image correlation (DIC) to simulate uniformity of strain transfer from matrix to wood fibers and found by addition of hybrid electrolyte wood fibres have increased homogeneity in HDPE. Besides, the hybrid addition enhanced flame retardancy of the composite that confirmed by LOI and heat release rate measurements [95]. Huang et al. (2020) followed a similar strategy, combined nanocellulose with polyethyleneimine (PEI) (a cationic polyelectrolyte), and with 25% addition of hybrid complex he reduced the total smoke release and heat release rate of WPC by 50.0% and 36.9%, respectively [96]. Battegazzore et al. layer by layer-functionalized rice husk with combination of branched polyethyleneimine (BPEI) and polyacrylic acid and then hot pressed the risk husk to form particleboards [97].

3.3.2 Particle Coating

Recently, several articles have been published investigating effect of particle/ nanoparticle coating on natural fibre reinforced composites. Sarker et al. introduced graphene oxide (GO, 1%) and graphene flakes (G, 10%) coating onto jute fibres in order to enhance mechanical and interfacial features of epoxy composites. Dip

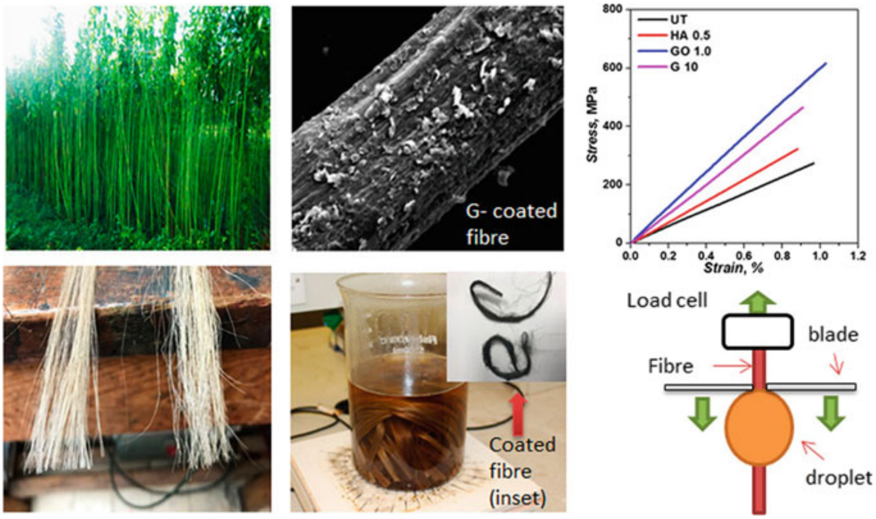


Fig. 8 Graphene (G) and graphene oxide (GO) coating on jute fibres improves mechanical strength of epoxy composites. Picture adapted from Ref. [98] with permission from ACS group

coating method was used to coat fibres and samples were analyzed by XPS and TGA. Then he made microbond test as shown in last image of Fig. 8 to reveal interfacial shear strength (IFSS) and tensile strength of treated and untreated jute fiber/epoxy composite. He found that the graphene materials coating onto jute fibres improved interfacial shear strength and tensile strength about 235% and 95%, respectively [98]. In 2019, same research group produced jute fiber reinforced epoxy composite by fiber individualization and hot water treatment and further coated with graphene oxide. They reported individualization of fibres combined with graphene-coating of jute fiber improves the elasticity and tensile strength of jute reinforced epoxy composites about 320% and 110%, respectively [99].

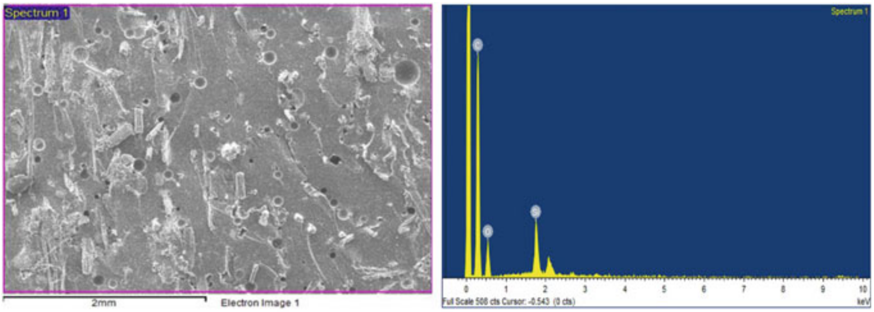
Wu et al. reported impregnation of Mg $(OH)_2$ to kenaf fibres and production of polyester composite. She improved modulus of rupture and tensile strength of treated kenaf reinforced polyester composite (MH13.3%-NFRC) by 73.9% and 54.6%, respectively, compared to controls. On the other hand, she stated that the specific bending and tensile strength are close or better than that of glass fiber reinforced polyester composite. Water absorption of the treated composites reduced more than 80% at 24 h compared to controls. More significantly, she investigated life cycle assessment of those novel composites and found that energy consumption was decreased by 33% compared to glass fiber composites and total environmental impact was reduced by 23% [56].

3.3.3 Natural Rubber Addition

Recently, there appeared an increasing interest on use of natural fibre reinforced natural rubber composites. The natural fibres as organic fillers have advantages as being biodegrade, high specific strength, low cost, abundance and so on [100]. However, the use of natural fibers and natural rubber in polymer blends can increase many properties of composites. Azammi et al. investigated physical and damping properties of alkaline treated kenaf fibre reinforced natural rubber/thermoplastic polyurethane (TPU) composites. Results indicated that composites of the kenaf fiber with increasing content of natural rubber absorb less water and have less thickness swelling due to good interfacial bonding between the fiber and the NR matrices. Dynamic mechanical analysis revealed that composites with high rubber content have better damping properties between other groups. Researches claim that 12.5 wt% of kenaf reinforced rubber/TPU composite has the potential to become a damping material as water resistance engine mount [101].

In 2021, Aiza Jaafar et al. reported the effect of the liquid natural rubber (LNR) on mechanical properties of epoxy/silica/kenaf hybrid composite. She revealed that up to 3 phr addition of liquid natural rubber enhanced flexural and impact strength by 22% and 43%, respectively compared to controls. SEM image and EDX analysis of fracture surface of composite proved well distribution of silane compound (Fig. 9).This phenomenon can be explained the LNR addition improved dispersion and adhesion of the additives [102].

Ferraira et al. found that carboxylated styrene butadiene rubber (SBR) coating of Curauá, jute and sisal fibers improved mechanical properties natural fiber reinforce cement composite [103]. Results of tensile test revealed that up to 70% increase of tensile strength was observed and pullout test confirmed that the SBR coating improved bonding strength more than 100% [103]. This method can be transferred to the polymer matrices.



(a) (b)

Fig. 9 **a** SEM image of fracture surface **b** EDX result of fracture surface of epoxy/silica/kenaf hybrid composite. Picture adapted from Ref. [102] with permission from Elsevier

3.3.4 Dopamine Treatments

Dopamine is a molecule containing catechol unit with a benzene ring with two hydroxyl side groups, and an amine group through an ethyl chain [104]. Dopamine treatment of natural fibres is one of the emerging methodologies that can be evaluated as the adhesion agent for natural fibre reinforced polymer composites [104]. Lin et al. treated bamboo fiber first with sodium hydroxide and/or further modified in aqueous dopamine solution (1 wt%) in order to reinforce PLA composite [105]. They found that flexural and tensile strength of the modified fibres increased about 16% and 35% respectively, where there is no significant difference between 4% alkaline + dopamine or only dopamine treated composites. On the other hand, thermal stability of the composites significantly enhanced by dopamine modification, while impact strength reduced for only dopamine treated composites, which additional alkaline treatment needed for better mechanical interlocking [105].

As shown in Fig. 10, Zhang et al. functionalized flax fibres by polydopamine and iron phosphonate in order to improve flame retardancy of natural fiber reinforced poly (lactic acid) (PLA) composites [57]. He stated that there is no improvement of fire retardancy (lower LOI and V-2 rating with UL-94 burning test); on the contrary, composites have worse thermal stability (TGA-DTG). However, the cone calorimeter test provided lower heat release rate (−16%) and lower total smoke production (−21%) compared to control. Besides, polydopamine and iron phosphonate treatment slightly reduced tensile strength of the composite, while tensile modulus increased considerably, which implies improved stiffness [57].

Brodowsky et al. used laccase enzyme to covalently bond dopamine to flax fibres in order to improve fibre-matrix interaction [106]. She observed change on surface

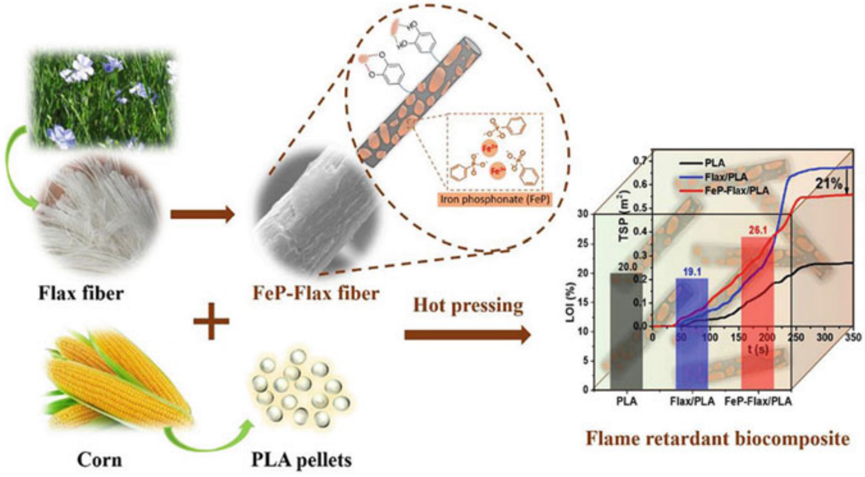


Fig. 10 Table of content image of polydopamine induced flax fiber reinforced PLA composites. Picture adapted from Ref. [57] with permission from Elsevier

morphology by SEM and AFM analysis. Tensile test of fibres showed there is no damage on the fibres due to treatment process and pull out test revealed that dopamine coupling improved the interfacial shear strength of epoxy composite by 30% compared to control.

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Morphology of the Interfacial Interface of the Natural Fibre Reinforced Polymer Composites

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1 Introduction

Technological advances in recent years have required the improvement of materials with advanced features and multifunctionality in order to satisfy the requirements for different applications. Therefore, a growing interest is observed in the use of composite materials, especially polymeric matrices, because of the versatility that these materials exhibit. This versatility is related to the possibility of combinations of different types of materials, where it is possible to vary the chemical composition, geometry, and form in which they are combined [1].

Composite materials have been successfully used in high-performance structural components used in aircraft, automobiles, ships, petrochemical plants, orthopedic implants, civil construction, eolic energy, and other applications [2–8]. Composite materials have demonstrated their efficiency, especially in technological applications that require a reduction in the weight of their components.

One of the biggest challenges at present is to make composite materials cost-effective and economically attractive. For this purpose, several manufacturing techniques have been developed and used in the composites industry, along with innovations in design, processes, and equipment. Moreover, the development of advanced materials for use as matrices, such as high-performance fibers, has enabled the achievement of composites with differentiated properties [1, 9].

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Composites are known to be materials consisting of a matrix and dispersed phase that is selected for their individual properties. However, it is also known that there is an interface in systems with distinct phases. When the mixing of these components occurs, their behavior will depend on the combination of the properties of the phases present, the structure of the composite, and its interface [1, 10].

In this context, one of the biggest challenges in the development of composite materials is to obtain an effective interaction at the interface reinforcement/matrix. In order to obtain the desired properties of a polymer composite reinforced with natural fibers, it is necessary to ensure that the mechanical load applied to the composite is effectively transmitted from the matrix to the reinforcement through the interface. Therefore, over the years several surface treatments have been developed to improve this interaction. These treatments aim to improve mechanical adhesion, electrostatic adhesion, chemical bonding, and even the interdiffusion of the surfaces [1, 11].

As the study of the matrix/stiffener interface in polymer composites reinforced with vegetal fibers is a topic of high relevance in Materials Science, several researchers have looked for different methods to improve the interfacial adhesion of these materials. In the Fig. 1 the evolution of the number of publications related to the topic in the last 20 years is presented, based in the SCOPUS platform. As search criteria, the keywords "morphology" AND "interfacial adhesion" AND "natural fiber" AND "composite" were adopted. The graph indicates an exponential increase in the number of publications, with a sharper increase after 2012.

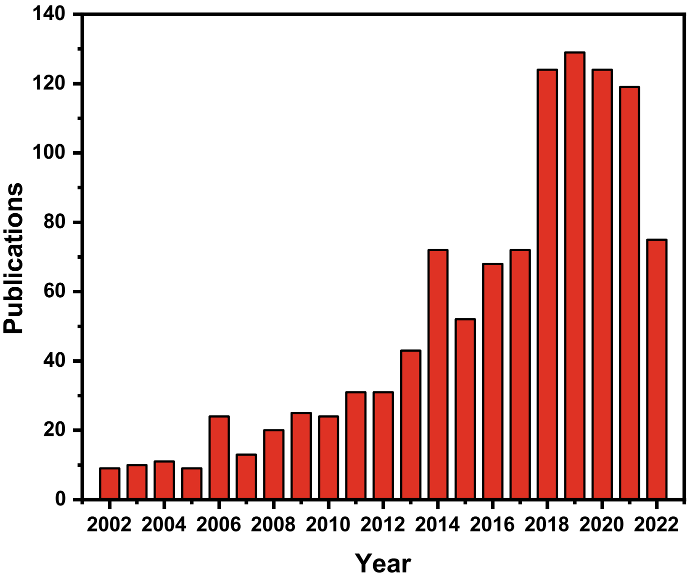

Fig. 1 Number of publications in the last 20 years in the SCOPUS platform on the topic of interfacial bonding in natural fibers

Given the importance of interfacial adhesion of polymeric composites, the objective of this chapter is to clearly introduce the concepts of interfacial adhesion of polymeric composites reinforced with lignocellulosic fibers and the morphology of the studied fibers. The structure of this chapter is divided as follows: first, the concept of natural fibers and the lignocellulosic elements that constitute them will be introduced, then the interface mechanisms between the fibers and the polymeric matrix will be discussed, and finally, the main surface modification techniques to improve the adhesion of vegetal fibers in composites will be discussed.

2 Lignocellulosic Fibers

Vegetable fibers are one type of natural fiber along with those with animal and mineral origin [12]. This type of material is also scientifically termed natural lignocellulosic fibers (NLFs) since they are mostly composed of cellulose, hemicellulose, and lignin, with other substances to a lesser extent, such as pectin and waxes. They are generally classified into five categories, fully related to their origin in the plant: seed, leaf, bast, fruit, and stalk fibers [13]. Table 1 presents different rates of the compositions of several existing fibers.

The NLFs have a large application in scientific research, given their availability and eco-friendly features [12]. Moreover, the lower cost of extraction of NLFs compared to the production of synthetic fibers has been drawing the attention of industries, as it does not require sophisticated technology or great production costs [2, 13, 66, 67]. On the other hand, the NLFs generally present a large range of values of mechanical strength, which are related to the heterogeneous chemical composition [66, 68–70]. Their composition is highly influenced by the weather, geographic location, season, and extraction methods [2]. Furthermore, the hydrophilic feature of these fibers is incompatible with the hydrophobic nature of polymers used in composite materials and requires further treatments that could compromise the cost-benefit ratio [66, 68–70].

Structurally, NLFs are arranged in sets of cells composed of long chains of cellulosic crystalline microfibrils connected in layers by the amorphous binder of lignin and hemicellulose [71, 72]. Thus, NLFs themselves are classified as a natural composite due to an amorphous matrix of lignin and hemicellulose reinforced with cellulosic microfibrils, forming a three-dimensional network, as shown in Fig. 2.

From Fig. 2, it is possible to notice the complex structure of an NLF. It consists of one outer primary wall and three inner secondary layers with helical-distributed microfibrils and a lumen inside. The primary wall presents a spaced and irregular network of compacted cellulosic microfibrils, while the secondary walls present a higher density of cellulose in the hemicellulose matrix. These walls differ in the orientation of the microfibrillar angle (MFA) and the cellulose-amorphous binder ratio. It is worth mentioning that the second secondary wall (S2) is the thickest layer of the microfiber, and, therefore, the most important in the NLFs structures as they

Table 1 Composition of the main NLFs present in the literature

Fiber	Cellulose (wt%)	Lignin (wt%)	Hemicellulose (wt%)	Pectin (wt%)	Wax (wt%)	References
Flax	60–81	2	14–21	2–5	1–2	[14, 15]
Hemp	57–78	3–13	11–22	1	0–3	[16–18]
Ramie	68–75	0.8–1.5	13–16	4–5	1–2	[19, 20]
Kenaf	45–66	14–20	12–20	0.4–2.7	0.3–3	[21, 22]
Guaruman	39–40	10–12	40–41	–	–	[23]
Jute	61–72	12–13	13–20	0.2	–	[24]
Sisal	67–78	8–12	10–14	10	2	[25, 26]
Cabuya	48–84	8.3–17	0.5–11	–	2	[27, 28]
Abacca	56–63	7–9	21	1–3	–	[27]
Betelnut	53	7	33	–	0.6	[29]
Banana	64–82	5–8.5	19	–	–	[30, 31]
Coir	32–50	30–45	0.15–15	1.8–4	–	[32, 33]
Bamboo	26–75	10–31	12–16	0.37	–	[34, 35]
Bagasse	32–55	19–25	27–32	–	–	[36, 37]
Sponge gourd	62	11.2	20	–	–	[36, 38]
Rice husk	38–45	–	12.2	–	–	[27]
Wheat straw	47–63	5.5–18.5	12–32	–	–	[39]
Oat	31–48	16–19	–	–	–	[27]
Napier grass	45–59	20–24	20–33	–	–	[40]
Curaua	73.6	9.9	5.5	–	–	[41, 42]
Henequen	60	8	25	–	2	[43]
Cotton	82–90	–	3	–	0.6	[27]
Nettle	72–84	2.2–7.5	6–12	–	–	[44, 45]
Pineapple	70–82	5–12	–	–	–	[25]
Hard wood	70–74	2.6–5.2	0.5–0.7	–	–	[46]
Soft wood	40–45	25–34	20–30	–	–	[47, 48]
Piassava	28	45	26	–	–	[27, 49]
Açai	45–47	31–34	10–15	–	–	[50]
Phormium tenax	67	11	30	–	–	[51]
Sansevieria ehrenberg	80	3.8	10	–	0.1	[52]
Sea grass	57	5	38	10	–	[27]
Isora	71–75	21–23	3.1	–	–	[53, 54]
Oil palm	60	11	–	–	–	[55]
Rachis	43–45	26	28–31	–	–	[56]
Rachilla	42	16	–	–	–	[27]

(continued)

Table 1 (continued)

Fiber	Cellulose (wt%)	Lignin (wt%)	Hemicellulose (wt%)	Pectin (wt%)	Wax (wt%)	References
Coconut	26–50	49–53	6–43	–	–	[57]
Barley	31–45	14–15	–	–	–	[27]
Pigeon pea	55	18	–	2.4	–	[58]
Arundo donax L	75.3	4.3	–	–	–	[59]
Rye	33–50	16–19	–	–	–	[27]
Esparto	42–44.5	12–17	25.6–27.5	–	–	[60]
Sabai	43–67	14–18	13–21	–	–	[61]
Phragmites communis	43–48	10–11	33–36	–	–	[62]
Coniferous	40–45	26–34	–	–	–	[27]
Deciduous	38–49	23–30	–	–	–	[27]
Cytostachys renda	42–49	17–22	19–23	–	–	[63]
Phychosperma macarthurii	39	18.2	19.1	–	–	[27]
Petiole bark	29–48	23–42	–	–	–	[64]
Kudzu	43–78	18–42	1–18	–	–	[65]

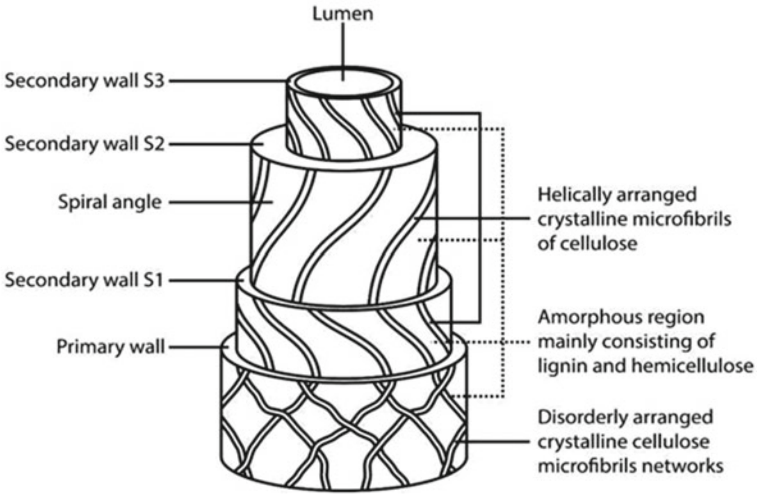

Fig. 2 Schematic representation of natural lignocellulosic fiber. Reprinted with permission from John and Thomas [72]

Table 2 The main chemical compositions of an NLFs

Composition	Characteristics
Cellulose	A natural polymer which makes up microfibrils. Although it does not vary chemically from fiber to fiber, its degree of polymerization is different for each NLFs, and this parameter is directly related to the mechanical property of the material [71, 73] Cellulose microfibrils are distributed helically. Its angle is different in each fiber and is also one of the determinants of the fiber's mechanical properties along with the volume fraction of cellulose [72]
Lignin	Natural polymer associated with cellulose that provides rigidity to the fiber. It has a hydrophobic character and acts as a cementing agent between cells, providing stress transfer between fibrils [72, 74]
Hemicellulose	Present at the interface of cellulose and lignin, acting as a cementing matrix between the cellulose microfibrils. It has a highly hydrophilic character and is soluble in alkaline solutions [73]
Pectin	A substance that makes up the physical structure and contributes to the flexibility of plants. It is only soluble in water after partial neutralization with alkali or ammonium hydroxide [11, 75]

are related to the mechanical property of the fiber [72]. Table 2 presents a brief description of the main compositions of NLFs and their roles.

3 Polymeric Matrices

In general, the matrices are responsible for the homogeneous external load transferring to the fibers. They give the final material environmental tolerance, durability, and surface appearance [76]. For the natural fiber-reinforced composites (NFRC), two types of polymers can be adopted as the matrix phase: thermosets and thermoplastics.

3.1 Thermoplastics

Thermoplastic polymers can be shaped in a viscous state and hardened by cooling [77], until solidification at room temperature. Under the heat, this resin can be reformed and reshaped without chemical reactions [76]. They are chemically inert and present high impact resistance and recyclability features as well as low processing cost. However, the processing temperature of the thermoplastics can occasionally degrade the natural fibers [9]. Thermoplastics with high melting temperatures cause chemical changes to the NLFs, such as depolymerization, dehydration, and oxidation [78]. The most common thermoplastic polymers used as matrices in NFRC are polypropylene (PP), polyethylene (PE), and polyvinyl chloride (PVC), polystyrene (PS) [9].

3.2 Thermosets

Thermoset resins are completely different from thermoplastics. They are cured by heat or a catalyst and cannot be reshaped or remodeled by heating [9]. Thermosets generally present a higher modulus, creep resistance, thermal stability, and chemical resistance than thermoplastic resins [76, 79]. On the other hand, this type of resin is brittle around room temperature and presents inferior fracture toughness as well as nonrecyclable characteristics. Nevertheless, they are the main choice as matrices of NFRC due to their superior mechanical properties and low processing temperature [78]. The most common thermosetting polymers used as matrix phase in NFRC are epoxy, polyester, and phenolic [9].

4 Nterface Mechanisms

Interfacial adhesion is vital for the mechanical behavior of composites [79]. The interface is placed in the interphase region between the matrix and the reinforcement phase, generally fibrous material, with dimensions varying between 1 atom to 1 μm thick, as presented in Fig. 3 [80, 81]. A strong interface forms strong bonds, transmitting the stress through the fibers and the matrix harmonically and uninterruptedly. Consequently, composites with stronger interfaces exhibit superior mechanical and thermal properties [82–84].

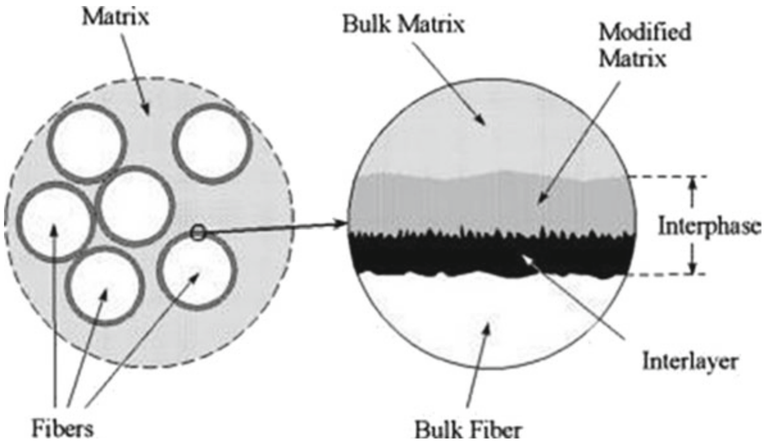

Fig. 3 Schematic illustration of a composite interface. *Source* Cech et al. [80]

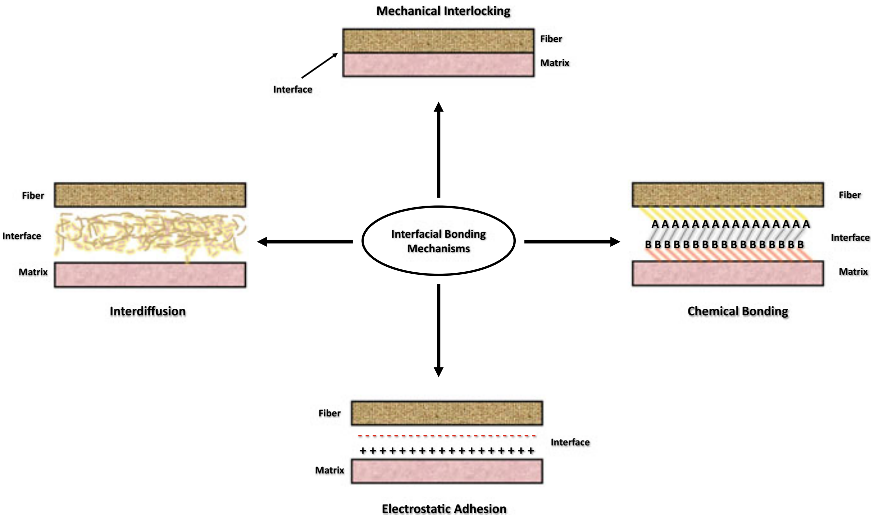

Fig. 4 Schematic of the principal interfacial bonding mechanisms

Figure 4 illustrates the main interfacial fiber/matrix bonding mechanisms. The main bonding mechanisms to promote interfacial adhesion in composites are electrochemical adhesion, electrostatic adhesion, mechanical interlocking, and interdiffusion. It is important to remember that interfacial adhesion is the result of the combined action of all these bonding mechanisms.

4.1 Mechanical Interlocking

Mechanical interlocking is a very typical adhesion mechanism that occurs between the rough surface of the NLF and the polymer matrix. Mechanical interlocking occurs through penetration of the molten polymer into micropores and defects present in the rough NLF surface. The roughness of the fiber creates a system that provides mechanical interlocking support without relying on electrostatic forces or chemical bonds, but only by anchoring the polymer to the fiber surface [81, 85]. Fiber surface roughness can be defined by the regularity (or roughness) of the valleys and clefts present on the fiber surface [86]. The higher surface roughness of the fiber, the greater the bonding area between the matrix and the fiber, increasing the bond strength at the interface region. Residual stress also influences the mechanical interlock. It is induced by the differential expansion or shrinkage of the two components of the composite [86]. Different from interdiffusion, which occurs on a nanometer scale, mechanical interlocking occurs on millimeter and micrometer scales.

In addition to already known surface treatments such as alkaline treatment, which remove components such as grease and waxes from the fiber surfaces, other techniques are used to increase the surface roughness of the fiber, such as the grafting of nano-sized components, e.g. nanoclays, nanoparticles, carbon nanotubes and graphene [87–92]. The addition of nano-sized grafts, which have a high surface area allows for an improvement in mechanical interlocking [93] developed hybrid composites of the epoxy matrix, reinforced with glass/kenaf fibers grafted with organomodified nano clay. The composites with the fibers grafted with nanoargyls showed higher flexural strength, as well as reduced cracking. Figure 5 shows the morphology of the composites with and without the addition of nanoclays, where it is possible to observe that the grafts increased the surface roughness of kenaf fibers, allowing better interfacial adhesion.

Prasad et al. [94] added titanium dioxide (TiO_2) nanoparticles in different concentrations on the surface of flax fibers and evaluated the mechanical properties and water absorption of epoxy matrix composites reinforced with these fibers. The TiO_2 nanoparticles promoted an increase in interfacial adhesion and mechanical properties of the fibers, besides promoting a considerable reduction in water absorption, an important characteristic since natural fibers are hydrophilic and polymeric matrices are hydrophobic. The micrographs in Fig. 6 show the influence of TiO_2 on the fibers after mechanical testing of the composites. The images show a severe pull-out of the fibers, the result of poor interfacial adhesion. The additions of 0.2 and 0.4 wt% TiO_2 resulted in a replacement of the fiber pull-out by fiber break out, and apparent good interfacial adhesion. Finally, the samples with 0.6 wt% TiO_2 showed poor interfacial adhesion, even though the authors indicated considerable improvement in mechanical properties and water absorption.

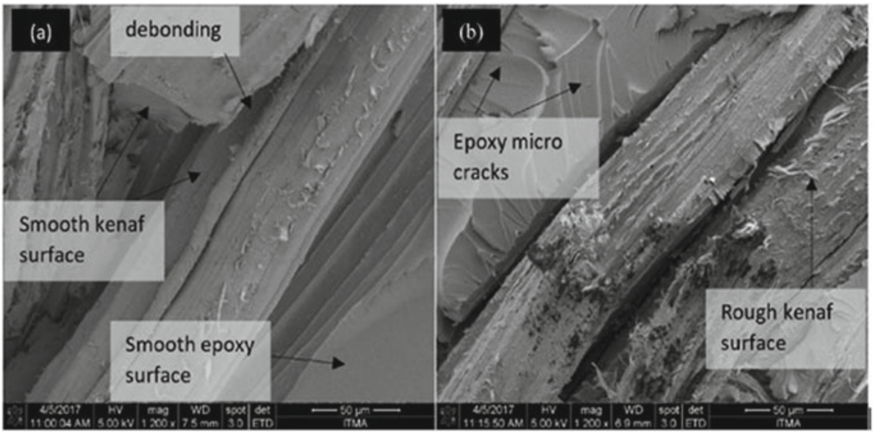

Fig. 5 SEM Micrographs of epoxy/kenaf/glass fiber composites: **a** unmodified with nano clay; **b** modified with nano clay. Reprinted with permission from Tay et al. [93]

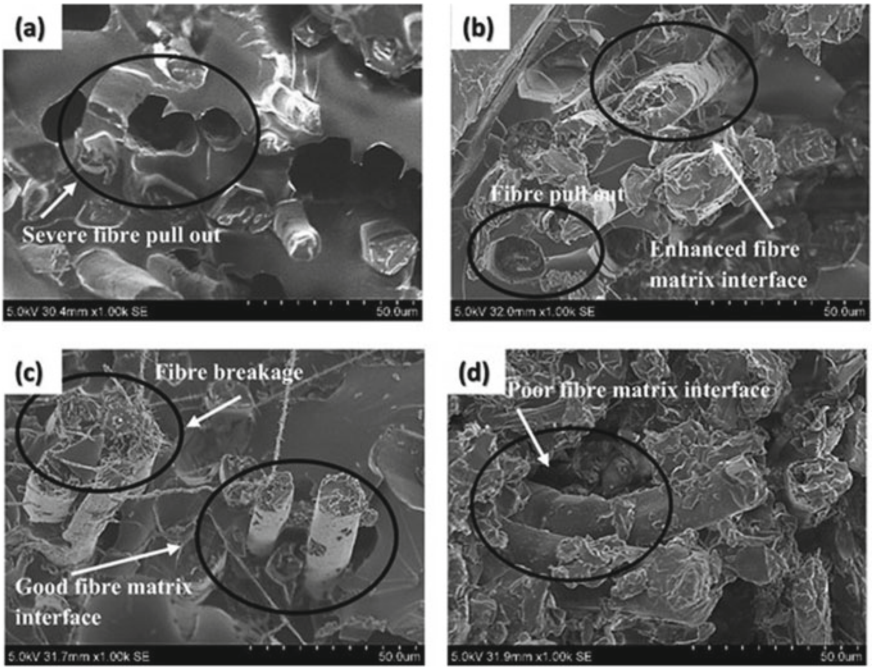

Fig. 6 SEM micrographs of epoxy/flax composites modified with different concentrations of TiO_2: **a** 0 wt%; **b** 0.2 wt%; **c** 0.4 wt%; **d** 0.6 wt%. Reprinted with permission from [94]

4.2 Physical Adhesion (Interdiffusion)

The physical adhesion mechanism is called interdiffusion. The interdiffusion is the result of a series of molecular interactions produced by Van der Waals forces, or hydrogen bridge between the molecules of the matrix and the natural fibers [86]. For a good interdiffusion at the interface, it is necessary to have good wettability, for this, it is necessary to check some characteristics such as surface areas and polarities of the composite components. The interdiffusion mechanism occurs in two steps: the first step requires an interaction between the matrix and the fiber, guided by diffusion and penetration. Then, after proper wetting, permanent adhesion of the constituents is realized through molecular attractions, via Van der Waals, electrostatic, and covalent bonds [95]. Although the concept of interdiffusion is attractive in the processing of composites, this mechanism can damage the fibers, and consequently reduce the mechanical properties of the composites. These materials, when exposed to high-temperature oxidation environments, are damaged due to the action of fiber debonding, pull out and fracture deflection mechanisms, damaging the interface of the composite [96]. Through interdiffusion, the molecules of the fibers, become entangled with molecules of the polymer matrix, as shown in Fig. 4. Under room temperature, molecular entanglement is affected. With increasing processing temperature, the polymeric chains in the matrix acquire higher energy for interdiffusion to occur. This energy allows the matrix chains to clear molecular motion. However, when the temperature reaches a range close to that of the glass transition, the molecular motion becomes so intense that it renders molecular entanglement ineffective [97]. Thus, the interfacial adhesion is reduced as entanglement is undone as a function of increasing temperature.

The polarity of the natural fiber also influences interfacial adhesion. The waxes present as constituents of natural fibers are nonpolar and have a lower surface tension than the other constituents of the fiber (lignin and waxes), which are polar. To change the polarity of the fibers and regulate the surface energy, alkaline treatments are used to promote greater wettability, creating permanent adhesion.

4.3 Electrostatic Adhesion

The electrostatic adhesion mechanism occurs as a result of the opposite charges of the composite interface surfaces, as observed in Fig. 4. Both surfaces have cationic and ionic charges, in which these opposing charges tend to bond [98]. The interfacial strength of composites is directly linked to the charge density of the interface components. Normally, the adhesion of composites is performed by chemical and physical adhesion mechanisms, where the interactions are made from Van der Waals and hydrogen bridges bonds, however, the bonding energy of these mechanisms is weaker, in the order of a few atomic radii [99, 100].

The electrostatic forces, on the other hand, provide greater resistance, in which their active distance is in the range of a few centimeters. Electrostatic adhesion does not occur in the usual way in composites reinforced with natural fibers, it usually appears together with physical and chemical bonds, thus there are few reports of electrostatic adhesion on natural fibers. Rajak et al. [101] propose that composites reinforced with NLFs be processed by the electrospinning method to develop materials with high interfacial energy. Currently, there are only reports of electrostatic adhesion in hybrid sandwich composites reinforced with synthetic fibers [102, 103].

4.4 Chemical Adhesion

The chemical adhesion mechanism is the best-known and most widely used mechanism to improve the interfacial adhesion of natural fiber-reinforced polymer composites. This type of adhesion occurs along the interfacial region when compatible chemical groups are added, allowing the matrix and fiber to bond. These modifications are made to both the matrix and the fiber to increase compatibility, as well as other aspects such as increasing hydrophobicity and improving mechanical strength. Figure 4 illustrates the chemical bonding mechanism, where an agent A or B is inserted into the interface region to promote interfacial adhesion. Currently, the literature presents a wide range of chemical treatments for natural fibers, which will be described in detail in the topic “Chemical Treatments”, however it is worth mentioning that the easy execution of these treatments makes them attractive for applications in composites. A well-known example of chemical treatment is the alkaline treatment, which removes the hydroxyl groups from the fibers, this removal causes the fiber to present hydrophobicity, repelling water and improving compatibility with the polymeric matrix [104]. Another treatment that influences the chemical bonding of composites is the addition of coupling agents, such as silanes and malleable agents. The silane agents interact with both the hydroxyl present in the fibers and the hydrophobic functional groups of the polymer matrix [105]. The treatment using coupling agents is performed in composites to enhance the performance of these materials depending on the environments in which it will be applied, such as environments with hygrothermal temperatures, or with UV rays incidence [106].

5 Natural Fiber Surface Modification

Natural fibers are hydrophilic, have low moisture resistance, and are incompatible with the polymer matrix, which is hydrophobic. This incompatibility of natural fibers results in a rough fiber/matrix interface, which reduces the mechanical features of the composites [107].

Therefore, natural fibers are subjected to surface treatments to change their surface and improve interfacial compatibility and adhesion between the matrix and the fiber. Because of the high concentrations of hydroxyl groups in hemicellulose and cellulose, plant fibers are polar and hydrophilic. These characteristics make plant fibers incompatible with hydrophobic and nonpolar molecules, leading to an inadequate interfacial connection between the two phases [74].

Depending on the type of treatment, changes can occur in surface morphology, physical and/or chemical structure, and hygroscopic properties. Several treatment methods aim to improve interfacial adhesion, but with the variety in chemical composition and the other characteristics of the plant fibers and characteristics of the various polymers, the effects of each treatment on the behavior of the composites will vary significantly. In general, the treatment will depend on the type of fiber, concentration and immersion time, temperature, experimental methods, and type of polymer matrix [108].

Some disadvantages of using natural fibers as reinforcement include weaker interfacial adhesion, decreased mechanical strength, moisture absorption, lower processing temperature (around 200 °C), lower durability and dimensional instability when compared to synthetic materials. In view of overcoming these aspects, some methods are proposed to treat and modify the surface of natural fibers, such as physical, Chemical, and biological processing. These techniques have different degrees of effectiveness and can improve the matrix/fiber adhesion, increase the mechanical properties as well as modify several unwanted characteristics for the final product [109] (Fig. 7).

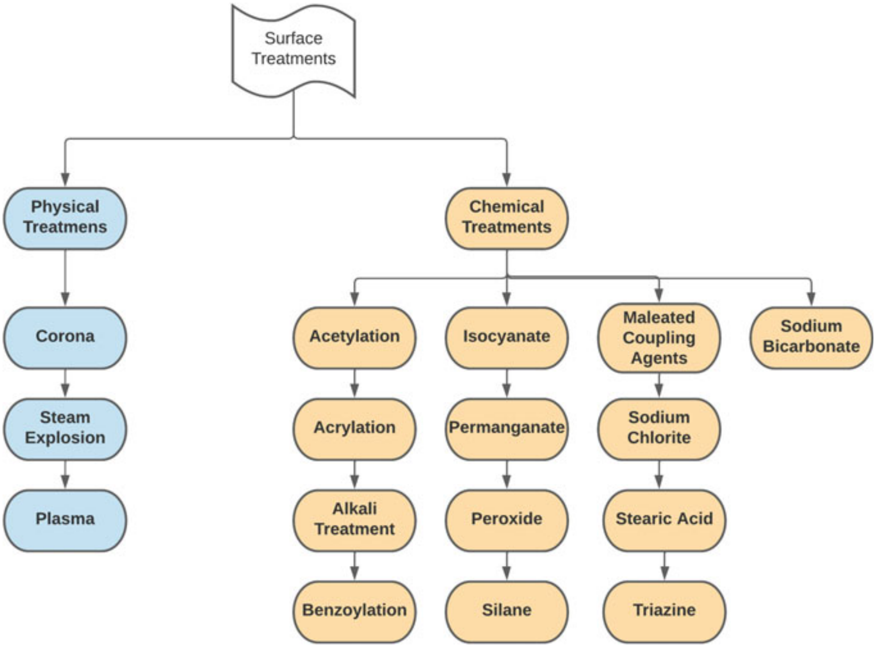

Fig. 7 Flowchart with the existing types of surface treatments. *Source* Authors

5.1 Effect of Surface Treatment on Water Absorption

The low adhesion between the matrix and the fiber causes void areas, and as a consequence, an increase in water absorption. To avoid these unwanted effects, certain treatments are used, such as fiber modification due to alkalinization, which reduces the fiber's moisture content. Potassium hydroxide treatments are commonly applied to reduce hydrogen bonding of cellulose and decrease the ability of OH groups to bond with water molecules. As a result, the hemicellulose is removed and its moisture content is reduced [110].

The water absorption causes the detachment between the fiber and the matrix due to the high susceptibility of the fibers to absorb it, causing a significant decrease in the mechanical properties of the composite. This occurs due to the polar and hydrophilic nature because of the existence of non-cellulosic components, such as lignin and hemicellulose, which promote water absorption due to the functional groups such as hydroxyl (OH) and carboxylic acids [79, 111].

5.2 Physical Treatments

Physical treatment is used to modify the surface of the fibers without interfering with their chemical composition. Natural fibers require treatments to improve their surface performance. Its advantages are an increase in the thermal, mechanical, and physical properties of the fibers. The chemical treatments are done to reduce the hydrophilic nature of the fibers, while the physical treatment, in addition to modifying the surface, increases the adhesion mechanisms and bonding strength between the surface and the polymer matrix. As a disadvantage, physical treatments are more expensive than chemical treatments [112, 113].

5.2.1 Corona

Corona treatment consists of subjecting the fiber to a specific electrical discharge for a relatively short time. This treatment can effectively promote fiber surface alteration, activation, and oxidation. It is well known that corona discharge has been shown to improve the compatibility of hydrophilic fibers with hydrophobic polymers [79].

The electrical discharges from corona treatment can be compared to the low-force electrical discharges that happen under atmospheric pressure [114]. This treatment is applied to activate surfaces, mainly polyolefins, increasing their surface tension. This treatment is applied to activate surfaces, mainly polyolefins, increasing their surface tension [115].

Fornari [11] explains the main challenge of this approach is the modification of three-dimensional objects, as corona discharges have well-defined directions and operating conditions to work on flat surfaces. The advantage of this treatment is the ability to alter the energy level, which in turn affects the potency of the corona process and, as a result, allows you to regulate surface activation levels. Furthermore, the processing time is very short, allowing high productivity and avoiding the production of undesirable by-products.

5.2.2 Steam Explosion

Steam explosion treatment uses high temperatures and high-pressure vaporization application, which causes a mechanical disruption of the material by an explosion in a collection tank. Using higher temperatures with more retention time will result in better homogeneous fibers and improve their wettability. This treatment reduces the ash and extractives and improves the tensile strength of the treated fibers [116].

This process transforms the lignocellulosic material into cellulose, amorphous lignin and hemicellulose. This process has some disadvantage, producing relatively short fibers and some composites may show inferior properties compared to other treatments [117].

5.2.3 Plasma Treatment

This treatment method is similar to corona treatment. This method induces changes in the surface of a material. This treatment can produce many modifications in the surface, which can vary depending on the nature and the type of the gasses used. Also, this method introduces surface crosslinking, produces radicals and reactive free groups, and increases/decreases the surface energy [79].

Studies have shown that regardless of the type of plasma used, the duration, and the specific conditions of the treatment, it is possible to modify not only the roughness of the fibers but also their hydrophobicity [118]. The most used gases for the plasma modification technique are helium, nitrogen, oxygen, and atmospheric air. One of the advantages of plasma treatment lies in the fact that the gaseous atmosphere can easily act on bulky or embossed surfaces. The treatment may be capable of introducing chemical groups on its surface, for example, carboxylic acid or ester. In addition, the physical roughness of the fiber can be increased, promoting a greater contact area with the composite matrix [11].

5.3 Chemical Treatments

Chemical treatments change the chemical composition, topography of the surface, and morphology of the fibers. Chemical treatments on natural fibers are necessary for:

I. remove non-cellulosic compounds;
II. improving the compatibility of the reinforcement with the matrix material;
III. improve the surface roughness of the fibers; and
IV. improve its thermal stability.

The chemical treatments have relatively low cost and ease of execution, so it is important to review the effects of different chemical treatments on the interfacial bond of fiber-reinforced composites [112].

Chemical treatments such as acetylation, alkaline, silane, benzoylization, and peroxide-based treatments improve fiber/matrix adhesion and reduce the hydrophilicity of natural fibers. [119] treated Agave Americana with NaOH, stearic acid, benzoyl peroxide, and potassium permanganate and obtained a significant increase in the tensile strength and strain of the samples analyzed. In addition, contact angle studies showed that the chemical treatments improved the roughness of the material, which promotes better wettability. Research has shown how composites of natural fibers in the epoxy matrix had an improvement in interfacial bonding, confirmed through morphology studies and pullout tests, reducing failure mechanisms (fiber detachment) and exhibiting better mechanical properties in dynamic and static tests than untreated fibers [120].

Sumrith et al. [121] conducted studies using NaOH and silane chemical treatments on water hyacinth fibers in bioepoxy matrix. The results showed an improvement in tensile, flexural, impact, hardness, thermal, dynamic properties, and surface morphology, which shows the effectiveness of chemical treatments in using natural fiber composites based on epoxy biopolymer, creating lightweight, sustainable, and stronger composites.

5.3.1 Acetylation

The acetylation treatment aims to modify the mechanical and thermal properties by plasticizing natural fibers. Acetic anhydride is the chemical commonly used in this in this treatment [122].

This treatment is carried out by the esterification reaction of the OH group of the fiber constituents (hemicellulose, lignin and amorphous cellulose) with the acetyl group (CH_3CO), acetyl groups are introduced into the cell walls of the fiber, replacing the OH groups, making the fibers with more hydrophobic properties and increasing the dimensional stability of composites. Thus, aiming to reduce its polarity, since the hydroxyl groups present in its main constituents are responsible for the polar character of lignocellulosic fibers. In this way, the fibers would become more compatible with non-polar polymers, forming more resistant composites [123, 124].

Bledzki et al. [125] treated flax fibers with acetylation and observed an improvement in moisture resistance and modification in surface morphology and an increase in tensile and flexural strength. Research has shown that acetylation-treated fibers with polyester matrices exhibited higher bio-strength and lower tensile strength than silane-treated composites. This treatment shows low resistance to UV degradation.

5.3.2 Acrylation

Acrylic acid ($CH_2 = CHCOOH$) is used to improve the interfacial bonding between fiber and matrix. $CH_2 = CHCOOH$ reacts with the fiber's cellulose OH groups and provides more access to the reactive cellulose macroradicals of the cellulose through polymerization [83]. The carboxylic acids of the coupling agents form ester bonds with the hydroxyl groups of the cellulose, reduce the hydrophilic OH groups of the fiber structure, and improve the moisture resistance properties. Also, grafting with acrylic acid to the matrix is initiated by peroxide radicals, the peroxide provides oxygen to extract hydrogen atoms from the tertiary carbon of the polymer chain. This coupling mechanism between fiber and matrix by acrylic acid increases the stress transfer capability at the interface and thus improves the properties of the composite [105, 126].

Research shows that this treatment increases tensile strength and reduces the water absorption of composites treated with acrylic acid. [127] Treated banana fibers with acrylic acid and with alkali treatments and observed that both treatments promoted an increase in the mechanical properties and moisture resistance of the composites, while the treatment with acrylic acid alone obtained lower results. The authors concluded that the combination of the two treatments promoted a significant increase in the flexural and impact strength of the banana fibers, as well as a decrease in the water absorption capacity. This treatment has the disadvantage of low resistance to UV degradation [128].

5.3.3 Alkali Treatment

Alkaline treatment can also be called mercerization, is the most widely used chemical treatments on natural fibers for reinforcing thermoplastics and thermosets. The important modification made by this treatment is the breaking of the H (hydrogen) bonds in the network structure, thus increasing the surface roughness. Also, this removes a certain amount of wax, oils and lignin that cover the outer surface of the fiber cell wall, depolymerizes the cellulose, and exposes the short-length crystallites [129].

This treatment removes, even partially, the hemicellulose, lignin, waxes, and fats from the fiber surface, promoting the formation of surface cavities that cause morphological changes in the lignin-cellulosic material. Thus, it can promote an increase in the cohesive force between the fiber/polymeric matrix besides the greater possibility of mechanical anchorage [126, 130].

According to Fornari [11], the alkali treatment may alter the physical condition of the fiber. The aspect ratio can be altered by the action of the NaOH solution. Decreasing the fiber thickness to increase the aspect ratio has a significant effect on the final properties of the composite.

Treating plant fibers with low concentrations of NaOH promotes controlled changes on the fiber surface, and results in more selective removal of constituents originating from the fibers. A more concentrated solution will act more aggressively in modifying the structure. The contact time with the solution is also a determining factor for surface modification, a relatively long contact period can completely destroy lignin, hemicellulose, and cellulose, causing total degradation of the plant structure [11, 128].

Setty et al. [131] concluded that there was a reduction in the hydrophilicity of *Limonia acidissima* shell powder through alkaline treatment, by FTIR analysis, and also confirmed the reduction of non-cellulosic compounds such as hemicellulose, lignin, and wax. Figure 8 shows a representative scheme of the alkaline treatment:

The alkali treatment has also proved useful in geopolymer composites reinforced with hemp fibers. Suwan et al. [133] investigated the feasibility of alkaline treatment in a geopolymer system, a decrease in compressive strength was observed due to the addition of the fibers, but a considerable increase in flexural and impact strength was obtained due to the improved adhesion between the natural fibers and the cementitious matrix.

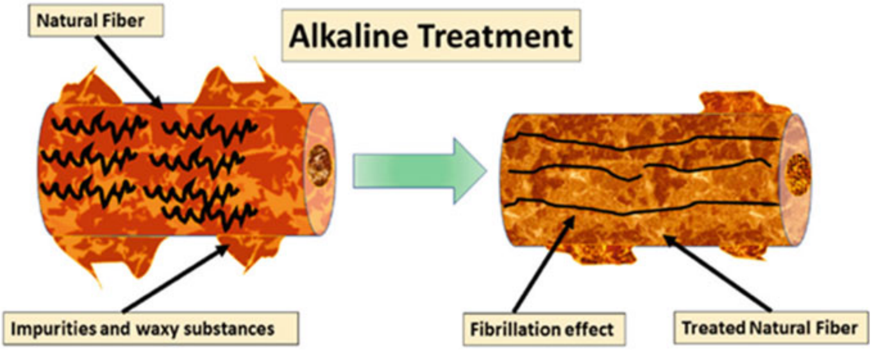

Fig. 8 Schematic representation of the alkaline treatment. Reprinted with permission from [132]

5.3.4 Benzoylation

Benzoylation treatment is used to change the hydrophilic character of natural fibers and is an important transformation in organic synthesis. Benzoyl chloride (C_6H_5COCl) has been used most often for the treatment, improving interfacial adhesion, increasing composite strength and also the thermal stability of the fiber [126].

The benzoyl groups ($C_6H_5C = O$) contribute to reducing the hydrophilicity of the treated fibers, increasing the compatibility of the fiber with the hydrophobic polymer matrix. The alkaline treatment of natural fibers in NaOH solution at appropriate concentrations can be done before adding benzoyl chloride. Benzyl chloride ($C_6H_5CH_2Cl$) can be used for benzoylation of natural fibers. The hydroxyl groups on the natural fibers can be replaced with benzyl groups ($C_6H_5CH_2$) during treatment, producing benzylated natural fibers [115].

Wang increased the interfacial bonding of the polyethylene matrix and the flax fiber by using an alkaline pretreatment of 10% NaOH and benzoyl chloride for 15 min and submerged the fibers in ethanol to remove the benzoyl chloride before rinsing with water and oven-drying. This treatment is also used to treat hemp fibers, showing a considerable increase in mechanical properties, dynamics, and reduction of water absorption [134].

The disadvantages of this method are fiber deterioration due to excessive chemical agents and benzoyl-treated fibers being highly susceptible to UV deterioration [128].

5.3.5 Isocyanate

Isocyanates are important binding agents used in composites with plant fibers. They can form permanent covalent bonds with the fibers, which definitely changes the surface conditions of the fiber [11].

The isocyanate acts as a coupling agent for fiber surface modification. The functional group ($-N=C=O$) of the isocyanate reacts with the OH groups of the lignin and cellulose of the fibers, and as a result, a urethane bond is formed. This chemical bond form strong covalent bonds between the fiber/matrix. Also, the isocyanate reacts with the moisture present on the fiber surface and forms urea that can further react with the OH groups of the celluloses. This secondary reaction results in higher moisture resistance properties of the fiber and provides better bonding with the matrix to improve the properties of the composite [105, 126, 135].

Sreekala et al. [136] concluded that this treatment has lower tensile strength and lower elastic modulus (E) when compared to permanganate peroxide and alkali treatments, but has better tensile strength and elastic modulus than silane and acrylic treatments. However, this method has low resistance to UV deterioration.

5.3.6 Permanganate

This treatment is performed by potassium permanganate ($KMnO_4$) in an acetone solution. This treatment forms highly reactive permanganate ions (Mn^{3+}) that react with the OH groups of the cellulose and form cellulose-manganate to initiate graft copolymerization. Also, this treatment improves the chemical interlock at the interface and provides better adhesion with the matrix [137]. The formation of cellulose-manganate is responsible for the higher thermal stability of the fiber. It also reacts with the lignin single bonds (hydrophilic OH groups) and separates from the fiber cell wall (reducing its hydrophilic nature) [105, 130].

Research has shown the result of using permanganate treatment on the coconut fibers. In addition to permanganate, the authors also used treatment with sodium hydroxyl, aiming to compare the modification of fiber roughness. Treatment with 5% sodium hydroxide produced a maximum rough surface of 3.96 μm. The treatment with potassium permanganate at 0.75% concentration it was possible to obtain a maximum roughness of 3.17 μm [138].

The disadvantages of this technique are that permanganate-treated fibers are vulnerable to UV degradation, $KMnO_4$ concentrations higher than 1% cause excessive delignification within the cellulosic structure and cause a degradation of fiber properties [139].

5.3.7 Peroxide

Peroxide treatment using organic peroxides has attracted attention in the treatment of natural fibers for the production of biocomposites due to the ease of processing and promotion better mechanical properties [140, 141]. Organic peroxides can easily decompose to release (RO) radicals, which react with the H atoms of the matrix polymeric chain and the fibers. The treatment is carried out at high temperatures after alkaline pretreatment. The use of high temperatures helps the peroxide molecules to break down easily [115].

Peroxide is a chemical compound with the functional group ROOR containing the divalent bonding ion (O–O). This method improves the adhesion between fiber and polymer matrix at the interface, reduces the water absorption capacity of the fibers, and enhances the thermal stability of the composites. Research shows that this treatment stimulates the adhesion of cellulose to thermoplastic polymers and facilitates processing, in addition to improving mechanical properties [142].

Sreekala et al. [136] studied benzoyl peroxide treatment on natural fiber-reinforced polymer composites, and observed an increase in tensile properties. The authors coated the fibers with benzoyl peroxide obtained by acetone solutions followed by alkaline pretreatment. Kaushik et al. [143] used peroxide to modify sisal fibers and observed that the surface of the treated fibers became rougher. Eliminating impurities in the fibers is an interesting strategy to increase the adhesion between fiber and matrix, improving both the interlocking mechanism and bonding reaction. Peroxide treatment has some limitations, such as decomposition and fiber damage at high temperatures, low strength, and UV deterioration [128].

5.3.8 Silane

Silanes are widely used to help in the bonding between fibers and polymeric materials, as they are able to form bonds between organic and inorganic materials. One of the most common silane compounds used for plant fibers is trialcoxysilane. The silane can modify the surface energy of the fiber making it more hydrophobic, and reducing water absorption [11].

This treatment can reduce the number of celluloses OH groups at the fiber/matrix interface. SiH_4 silane-binding agents are used to reduce micropores on the fiber surface. This treatment reduces the OH groups of the fiber, promoting a better interface. The OH groups of the fiber react with the silanol and form a covalent bond with the cell wall, preventing fiber swelling by forming covalent cross-links [144].

Research shows that some fibers show better results with silane treatments than with alkaline treatments, for example, oil palm fiber. However, this technique has some disadvantages, such as alteration of the intrinsic structure of the natural fibers by excessive chemical solutions, and also has low resistance to UV degradation [145].

5.3.9 Maleated Coupling Agents

This treatment is used to treat the surface of the fibers and the matrix, resulting in a better interfacial bonding and increased mechanical properties. The maleic coupler forms the bond (C–C) in the polymer chain with the matrix. Better bounding can be formed between the coupling agent and the fibers when this treatment form covalent bonds, increasing the molecular bonding between the polymeric matrix and the maleated coupling agent. This method proper a interaction with the surface functional groups of the fiber with the polymeric matrix, forming carbon–carbon bonds between the fibers and the matrix [146].

Mohanty et al. [147] Used malted coupling agents to modify jute fibers, and compared it with untreated fiber, and observed an increase in tensile, flexural, impact strength. The best results obtained were for the composites with 30% by volume addition of treated jute fibers.

This method has some disadvantages, the treated composites have a high adhesion of fiber/matrix, resulting in fiber disintegration by tearing, and they have low resistance to UV deterioration [128].

5.3.10 Sodium Chlorite

Several works involving sodium chlorite (NaCl) have been developed and have proven useful to treat the surface of natural fiber-reinforced composites. The fibers are bleached in an acidic sodium chloride solution, such as $NaClO_2$, delignification of the lignocellulosic compounds occurs. This method is useful for increasing their hydrophobicity, as well as increasing the flexibility of the fiber. Research shows that this method reduces the hardness, making the composite more flexible, besides promoting the increase of mechanical properties, such as bending limit [130].

Nabinejad et al. [148] treated oil palm fiber with sodium chlorite, and reduced the fiber diameter from 250–350 μm to 10–15 μm. Mechanical tests showed the superiority of the sodium chloride-treated fibers compared to untreated fibers. The disadvantages of this treatment are that the delignification reduces the tensile strength, in addition, this treatment has low resistance to UV degradation.

5.3.11 Stearic Acid

The treatment is done using stearic acid ($CH_3(CH_2)_{16}COOH$) in ethyl alcohol solutions to treat the natural fibers. Linen fibers showed higher crystallinity when compared to untreated fibers, showing that stearic acid decreased the fiber surface free energy. Sugarcane fibers treated with stearic acid had reduced moisture content and improved mechanical properties, mainly impact resistance, with an increase of about 144–200%. Stearic acid has the following functions [149, 150]:

1. Increases compatibility between fiber and polymeric matrix;
2. Increases the melt flow, reducing the viscosity.

As a disadvantage, this technique removes the non-crystalline components of the fiber, modifying the fiber's topographic surface [139].

5.3.12 Triazine

This treatment reduces the number of free hydroxyls that would bind to water, through the news covalent cross-links from matrix/fiber, reducing the swelling and the hydrophilicity from the surface of the fiber. The reaction of the triazine ($C_3H_3N_3$)

start with the hydroxyls groups from the cellulose, enhancing the interfacial bonding of the fiber/matrix and provides a higher resistance of the moisture. The composites treated by these techniques show increased mechanical strength, better thermal properties and higher humidity resistance. In addition, this treatment has a low resistance to UV degradation [130].

5.3.13 Sodium Bicarbonate

The treatment with sodium bicarbonate solutions is slightly alkaline, due to the formation of carbonic acids and hydroxide ions interacting with the fiber surface in the same way as the traditional mercerization treatment. Sodium bicarbonate reduces the hemicellulose and lignin content [151].

Fiore et al. [152] treated jute fibers and flax in an epoxy matrix with 10% sodium bicarbonate and found that the treated composites obtained a decrease in moisture absorption, an increment in flexural and tensile strength after the aging process of 2 months in salt spray exposure, besides the jute fibers presented a significant increment in impact resistance of about 300%, while the flax fibers had an increase of 61%. Figure 9 shows the SEM images of surface of composites after the salt spray exposure.

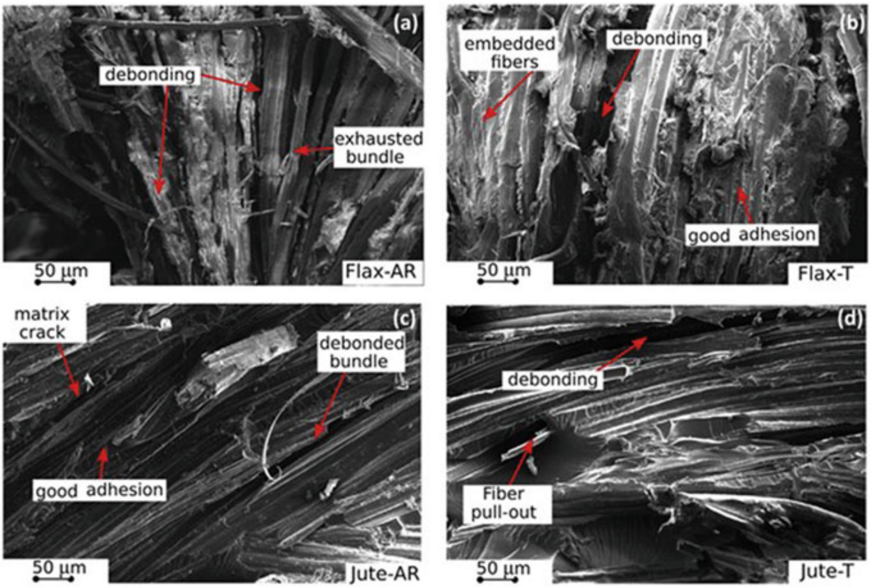

Fig. 9 SEM images of the fracture surface of laminate composites after salt-fog exposure. Reprinted with permission from [152]

6 Conclusions

This chapter presents an overview of the interface of natural fiber-reinforced polymer composites and their morphology. The interfacial adhesion of composites is influenced by a variety of factors, such as fiber composition, the type of polymer in the matrix phase, interfacial adhesion mechanisms, and surface treatments employed. NLFs have a series of constituents in their composition, which have a direct effect on characteristics such as crystallinity and interfacial adhesion, thus, it is important to know these components in order to use a surface treatment in the correct way. The type of polymer used in the matrix influences interfacial adhesion, since the processing method of the composite can determine the wettability of the polymer, which can improve or worsen the properties of composites. Finally, the different types of interfacial adhesion mechanisms allow the choice of a processing technique or surface treatment to improve composite properties by strengthening the fiber/matrix interface.

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Spectroscopic Analysis of Interfacial Adhesion in Natural Fibre Polymer Composites

Shruti Swaroop Pattnaik, Ajaya Kumar Behera, and Nigamananda Das

Abstract The ideal substitute materials for plastic to address the issues of pollution and non-biodegradability are natural fibre reinforced composites. These are non-toxic, biodegradable, less expensive, and have potential applications in a variety of industries, including packaging, furniture, and home goods. This is because natural fibres are abundant in nature, biodegradable, and inexpensive. However, due to inadequate interaction/bonding between hydrophilic natural fibre and hydrophobic thermoplastic/thermoset matrix, its competency with thermoplastic is hampered by low mechanical qualities and other physical features. Numerous studies have been conducted with the goal of strengthening the chemical interaction between the fibre and matrix, either by roughening the surface of natural fibre or by altering resin. Spectroscopic examination, including Fourier transform infrared, Raman spectroscopy, nuclear magnetic resonance, and X-ray photoelectron spectroscopy are used to characterize the produced composites after modification of natural fiber. The strength of the composites in question is provided by the connection between functional groups of fibre and resin at the interface, which was discovered through spectral analysis of composites.

Keywords Natural fiber · Biodegradation · Physical properties · Spectral analysis · Biodegradation

1 Introduction

Natural fibres are viable options for reinforcing materials because of their physical–mechanical characteristics, affordability, and biodegradability. Compared to synthetic fibres, natural fibres made from cellulose have a lower density and are also more rigid and durable [1]. Compared to synthetic fibres, natural fibres are less expensive and hazardous. Flax, hemp, jute, kenaf, and sisal are the quite significant

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natural fibres utilized in composite materials. All of these natural fibres are composed of typically long cells with cell walls that are rather thick, giving them their stiffness and strength. The cells are arranged into long, thin threads in the majority of plant fibres. The roughness, cell length, strength, and stiffness of the natural fibres vary from one another. Since cellulose and lignin make up the majority of these fibres, they are referred to as lignocellulosic fibres [2]. For the manufacture of composite materials using thermoplastic/thermoset resin, a number of different natural fibres are utilized as reinforcement or fillers is given in Fig. 1 [3] and their photographs are given in Fig. 2 respectively.

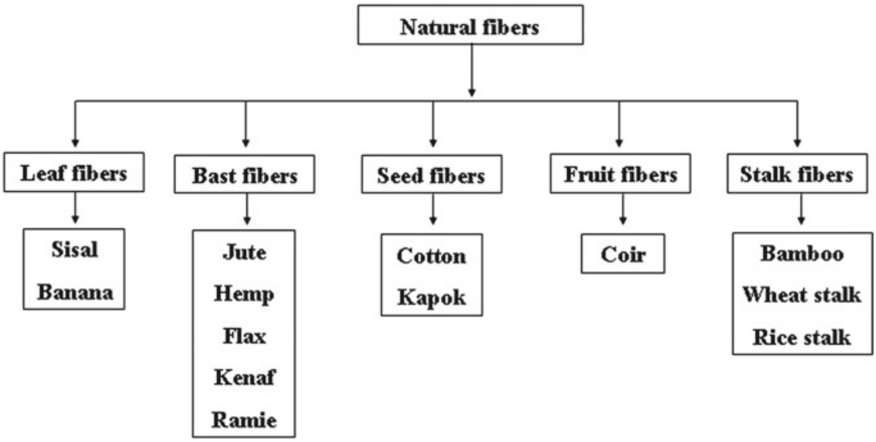

Fig. 1 Types of natural fiber use for composite fabrication

Fig. 2 Digital photographs of natural fiber

1.1 Polymer Reinforced Composites

A composite is a two-material mixture in which one of the components, referred to as the reinforcing phase, is present as fibres, sheets, or particles that are incorporated into the matrix phase of the other material. Metal, ceramic, or polymer are all acceptable choices for the matrix and reinforcing materials. Due to their great strength and low density, fibre reinforced polymeric composites have found extensive use in structural applications where they significantly outperform traditional metals. Polymers are the most often utilized matrix materials for composite fabrication. For many structural purposes, the mechanical characteristics of polymers are generally insufficient. Their stiffness and strength are particularly weak when compared to those of metals and ceramics [4]. By adding polymers to other materials, these issues are resolved. Second, fabricating polymer matrix composites can be favourable at low temperature and low pressure too. In addition, simpler machinery is required to create polymer matrix composites. As a result, polymer matrix composites were created quickly and immediately became popular for structural applications. There are two categories of polymer composites:

1.1.1 Particle Reinforced Polymer Composite

Particle composites consist of particles of one substance spread in a polymer matrix of a second material. The majority of particles are spherical, ellipsoidal, polyhedral, or irregular in shape, though they can be any size or shape.

1.1.2 Fiber Reinforced Polymer Composite

Typical composites with fibre reinforcements, are made of a matrix and synthetic/natural fibres. The fibres serve as support and the strengthens the composite, while the matrix binds whole of the fibres collectively and distributes the impeding stress among the reinforcing fibres. Composite materials may occasionally have filler added to them to improve the manufacturing process, give them distinctive characteristics, or reduce the cost of the end product. Among the main constituents of natural fibres derived from various plant sections are cellulose, hemicellulose, lignin, etc. Table 1 lists the main elements of certain natural fibres [5, 6].

When a polymer serves as the matrix material and natural fibres as reinforcement, the composite is termed to as a natural fiber reinforced polymer composite (NFPC). The goal of NFPCs is to create new custom-made characteristics by utilizing the superior qualities of the reinforcements and matrix. The main considerations in the fabrication of high-strength composites are the mechanical properties of reinforcements. The degree of mechanical qualities of natural fibres is influenced by a number of variables, including fibre orientation, crystallinity, moisture content, and fibre flaws. Tensile properties of various cellulosic fibers are given in Table 2 [6].

Table 1 Composition of natural fiber

Natural fiber	Origin	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Moisture (%)	Wax (%)
Jute	Bast	59–65	12–13	11.8–12.9	1–3	8–12	0.5
Banana	Leaf	63–67	12–15	5–8	3–5	10–12	–
Hemp	Bast	67–75	16–18	2.9–4.8	1–2.5	6–10	0.8
Sisal	Leaf	47–62	21–24	7–9	3–4	10–15	2
Cotton	Seed	90–93	3–5	–	1–2	7–8	0.6
Coir	Fruit	42–57	10–12	42–27	3–5	5–8	–
Flax	Bast	62–71	16–18	2–2.5	2–3	8–12	1–2
Ramie	Bast	65–78	13–18	1–2	2–3	7–15	0.3–1
Pineapple leaf fiber	Leaf	70–80	10–13	10–15	1–3	–	–
Kenaf	Bast	50–57	15–18	8–13	2–4	–	–

Table 2 Tensile properties of some natural fibers

Fiber	Diameter (μm)	Density (g/cc)	EB (%)	TS (MPa)	TM (GPa)
Jute	25–200	1.4	1.5–1.8	393–773	26.5
Flax	40–600	1.5	2.7–3.2	345–1035	27.6
Hemp	25–500	1.47	1.6	690	70
Ramie	–	1.5	3.6–3.8	400–938	61.4–128
Sisal	50–200	1.4	2.0–2.5	511–635	9.4–22.0
Coir	100–460	1.2	30.0	175	4.0–6.0
Cotton	12–38	1.5	7.0–8.0	287–597	5.5–12.6
Pine apple	20–80	1.4	1.6	413–1627	34.5–82.5
Palm	240	1.2	17	98.14	2.22

EB: Elongation at break, TS: Tensile strength, TM: Tensile modulus

1.2 *Interfacial Region Between Natural Fiber and Polymer in Composite*

The fibre reinforcement in the composite has two major functions; (i) bearing the composite's tensile load while spanning the cracks in the matrix and (ii) limiting spread of the fissure by releasing energy surrounding the point of fracture [7]. The process starts with a stress-induced deformation of the matrix that induces shear stress in the interfacial bonding region. To compensate for this tension, the tension in the fiber is released. Microcracks appear on the substrate and propagate as soon as the strain energy neighboring the fissure angle is greater than the aggregate energy required fabricate a novel surface. When the fibers bridge the cracks in the matrix, pressure carried by the matrix are transferred to the fibers through the interface. The strain energy is degenerated near the tip through interfacial failure (fiber tearing, interfacial separation, etc.) and fiber breakage, thereby preventing crack growth. Since the interface is a common boundary, once a failure occurs, it is impossible to achieve fiber reinforcement to the matrix [8].

Interfacial regions are more prone to stress concentrations than the fibers or matrix, leading to the formation of microcracks that reduce the load-carrying capacity of NFPC. Moisture breaks the interfacial bonds by weakening the chemical interactions and mechanical inter-adhesion at the fiber-matrix interface, moisture can potentially disrupt the interfacial linkages. Prolonged exposure to this moisture causes microcracks to propagate, creating supplementary conduits for moisture to diffuse, thereby fast-tracking the failure of the fibers and matrix, as well as the interfacial bonds between them. Synthetic fibers are slightly less hydrolyzed as compared to natural fibres; hence the interfacial regions aren't not a necessity for them [7]. A proper adhesion between the components of the NFPCs is also affected by:

1.2.1 Bonding Layer

Greater bond strength results into favorable interface and vice versa. Extension of the matrix crack causes the interface bonding to relieve the strain concentration at the facade of the matrix crack, which in turn, prevents fiber detachment and deformation of the fissure and extension into the fiber [9].

1.2.2 Load Transfer

Interfacial adhesion must be adequate, not too much strong to make the composite brittle and not too less, which tends to debond under low tension, thereby reducing the mechanical character of the composite. Stronger adhesion provides a less dampening effect. Strong shear strain and an energy-dissipating substance are both needed to make such a composite. According to [10] in engineering structural applications, a fiber-reinforced composite with low dampening properties is an economical way to increase energy dilution. Increasing the matrix volume % at the expense of stiffness and strength has been proposed as a way to improve dampening.

1.2.3 Physical–Chemical Compatibility

Krishnasamy et al. [11] correlated physical–chemical compatibility with the respective coefficients of thermal expansion of the fibre and matrix to prevent a divergence in their nature, hence improving its adhesion. Coatings on the fibres may establish a buffer zone between them and the matrix.

1.2.4 Prevention or Inhibition of Oxidation

Elevated temperature during fabrication creates an oxidizing environment which impacts this boundary in between. The features of natural fibre in the composite are greatly diminished as a result. As per [12], erosion brought on by an oxidizing environment must be prevented in addition to controlling atomic diffusion and chemical reactiveness between the fibre and matrix. The cellulose is probably degraded by low thermal stability. According to Yao et al. [13], the majority of natural fibres thermally degrade by 60% between 215 and 310 °C, with an apparent activation energy of 160–170 kJ/mol.

1.2.5 Appropriate Thickness

A proper thickness of the interfacial bond must be maintained; which can be clearly studied through various micromechanical testing methods and SEM.

1.3 Mechanism of Fibre-Matrix Interaction

Drzal M Madhukar [14] and Avancha et al. [15] have stressed how the effectiveness of load transmission is influenced by molecular interactions at the interface as well as the thickness. Fibres in NFPCs can be categorised into two regions- one of the region which doesn't interact with the matrix directly hence, faces no change in characteristics, and other part which is antagonistic to this. The handling of material that has been adsorbed on the fibre or matrix before the production of the composite is where the first change in properties may be found. The initial surface of the fibres can be altered by adding or removing functional groups, making the original fibre no longer be relevant. Scanning electron micrograph of jute fiber can be observed in thermoset soy matrix at the interface in Fig. 3 where jute functional groups is chemically bonded with that of the soy matrix [16].

Also, being subjected to air for a long time can alter or eliminate the reactivity of the fibre or the matrix, since some chemical species from the air may also adsorb on this surface. The second basis of adhesion is the interdiffusion or chemical reactions between the fibres and the matrix. Drzal et al. [17] commented on the interface formation in composites, stating that it can occur between electrically neutral species by physical attraction, molecular entanglement, inter-diffusion of elements, mechanical interlocking and chemical bonding as weak as Van der-Waal's forces.

1.3.1 Mechanical Interlocking Mechanism

Based on a polymeric matrix, surface alterations can be made chemically using techniques including alkali treatment, acetylation, silane treatment, cyanoethylation,

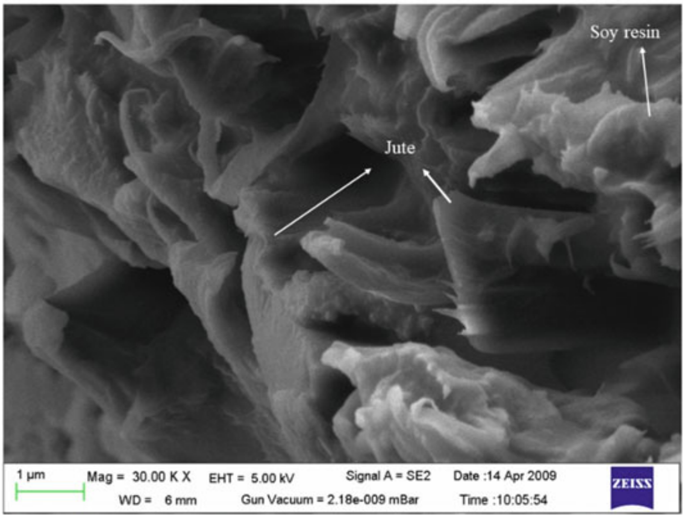

Fig. 3 Scanning electron micrograph of jute in soy composite

grafting, esterification, etc. For, interlocking the fibres with the matrix, we need to create a rough surface on both of them. Grafting on fibres can increase the roughness of the fiber surface. Graft polymerization with acrylonitrile on jute and pineapple surface enhanced the fiber properties by reducing moisture absorption [18]. Alkali treatment also increases the roughness as it removes the fat and waxy layer of the fibres, hence exposing the rough surface. Thermoset polymers being non-polar and hydrophobic in nature are not compatible with the hydrophilic natural fibres. As a result, they showed poor adhesion properties.

1.3.2 Electrostatic Adhesion Mechanism

The opposing charges that develop on the surface of the fibres and matrix are what cause the electrostatic binding to happen. The strength of the interfacial adhesion here depends on the charge density [19]. Electrostatic discharge treatments have resulted in increased adhesion and can also adsorb particles which may also affect the surface attachment. Hence this form of adhesion is also an important feature to broaden the applications of NFPC.

1.3.3 Chemical Bonding Mechanism

Chemical bonding functions through inter-diffusion mechanism. This takes place via 2 phases: adsorption and diffusion. Firstly, close interference between fibre and matrix occurs via ‘spreading’ and ‘penetration’. Permanent adhesion is formed after wetting, by molecular attractions like Vander Waal, covalent and electrostatic. A good wetness also results into interdiffusion. This can be enhanced by the use of coupling agents which have 2 different functional group ends, with one end reacting with fibres and other coupling with the matrix. For example, for silane, one end reacts with the -OH of the natural fibres, and other end reacts with the matrix. Appropriate coupling agent is chosen based on the operating conditions of NFPCs [20].

1.4 Surface Modification to Improvise Inter-Facial Interaction

1.4.1 Alkali Treatment on Fiber Surface

Natural fibres are frequently chemically modified to address their flaws. To augment the characteristics of jute reinforced materials, [21] proposed surface treatment of jute using either an alkali or a polymer. Jute fibres have undergone an alkali treatment to enhance the mechanical and wettability of the resin. By separating fibre into fibrils, alkali treatment eliminates hemicellulose and other soluble carbohydrates. Following alkali treatment, the fibres begin to fibrillate, increasing the effective available surface area for interfacial connection between the fibre and matrix. Jute's cellulose hydroxyl group becomes liberated after alkali treatment, ultimately increasing the fiber's strength and modulus [22]. After jute fibres were treated with alkali, [23] found that the hemicellulose had been removed. Additionally, they hypothesised that jute's inter-fibrillar area is likely less stiff and dense, allowing for fibrils to reorganise themselves in a way that favors tensile deformation. Figure 4 illustrates schematically how alkali treatment releases bound cellulose groups from natural fibre.

After mild alkali treatment, hemicellulose and lignin from natural fibre were reported to have been partially removed by Mwaikambo and Ansell [24]. According to [22] and [25], higher concentration alkali treatment (17.5%) decreases strength and modulus properties of natural fiber due to elimination of lignin, whereas mild

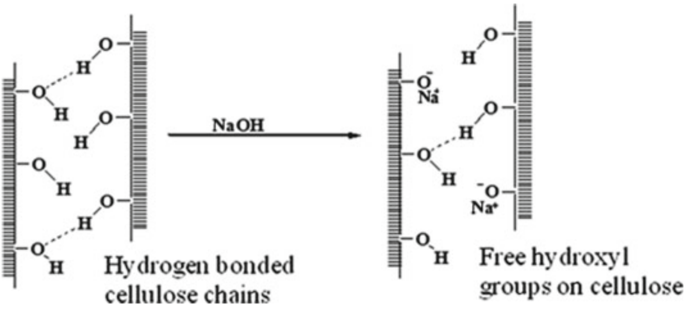

Fig. 4 A schematic presentation of alkali treatment on natural fiber surface

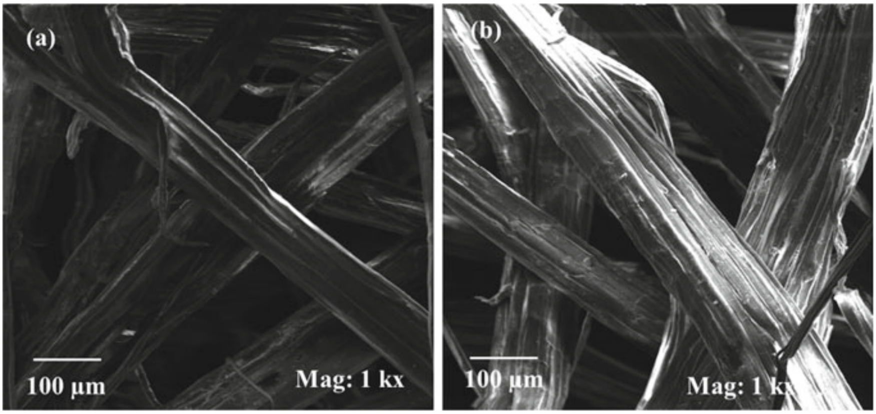

Fig. 5 Scanning electron micrographs of **a** raw jute fiber, and **b** alkali treated jute fiber

alkali treatment can increase the strength of sisal, coir, hemp, banana, pineapple, abaca, and coir fibres [26, 27]. This is because there are now more places where fibrils can mechanically interlock because of interfacial adhesion between the resin and fibre contact. Figure 5 depicts scanning electron micrographs of the natural fibre jute, where we can see that the raw jute fibre surface (Fig. 5a) broadened and fibrils emerged following mild alkali treatment (2% NaOH for 2 h) (Fig. 5b).

1.4.2 Esterification

Malleated polypropylene (MAPP) or malleated polyethylene (MAPE) copolymers are extensively used in industry as commercial coupling agents to enhance the interface between cellulosic fibres and thermoplastic matrices [28, 29]. All of these were premeditated through various spectroscopic methods. Surface modification of jute using esterification by alkenyl succinic anhydride [30] have been well explored in jute-polypropylene composites. Keener et al. [31] looked at the impact of several MAPP coupling agents on PP composites augmented with flax and jute. They discovered that the inclusion of a compatibilizer resulted in a 41% improvement in the tensile strength of composites reinforced with flax fibre.

1.4.3 Silanization

Silane treatment of lignocellulosic fiber is through various silane coupling agents. Silane acts as bridge between hydrophilic fiber and matrix by forming a bond with cellulose at temperatures above 70 °C [32]. The fundamental benefit of utilising silane coupling chemicals is that one end of them has alkoxysilane groups that can react with the -OH-rich fibre surface, while the other end may be tailored to fit a particular matrix. Jute-PP composites that had been silane treated had improved tensile and dynamic mechanical characteristics. Jute fibers were broken without complete pullout and PP matrix remained surrounding the fibers when fracture surface of composites was observed. Silane treatment of the jute fibers increased the fiber-matrix interaction through a condensation reaction between hydrolyzed silane and hydroxyl groups of jute cellulose [33].

1.4.4 Enzyme Treatment

The majority of the non-cellulosic elements that cover the raw natural fibres make them hydrophobic and increase the surface's non-wettability, which leads to weaker adhesions. Micro-organismic enzymes, such as lipases and hemicellulases, were shown to be able to remove this extra layer and alter the surface, hence enhancing the adhesive forces between the fibre and the matrix. Pectinases, hemicellulases, laccases, and cellulases are the principal enzymes necessary for the enzymatic extraction of natural fibres, with xylanases being the most important. Pectin is hydrolyzed by pectinases, which causes the fibres and non-fiber components to be separated [34].

1.4.5 Gamma Radiation Treatment

Gamma radiation is a very potent ionizing radiation that has the ability to change a material's internal structure and reduce its hydrophilicity, which improves crosslinking between the natural fibre and matrix. After receiving gamma radiation treatment, jute-polypropylene composite was examined by Khan et al. [35]. Hoque et al. [36] investigated how gamma radiation affected a bleached natural fiber-polyester composite. When employed as a matrix part for any composite material, polyester resin exhibits high levels of cross-linking and structural alterations on the surface as a result of gamma radiations, according to Martinez-Barrera et al. [37] 's study of this impact.

1.4.6 Plasma Treatment

This method is economical and ecologically responsible. It activates the surfaces of synthetic/natural fibers and wood. It increases the wettability which in turn increases the binding strength, adhesion and hydrophilic properties of the material. As compared to chemical activation, the process is efficient and commercially feasible [38], environment friendly since it is solvent free, modifies surface properties without impacting bulk properties. Anwar and Bhuiyan [39] carried out low temperature plasma treatment of jute surface and observed disrupted fiber surface due to physical sputtering and chemical etching. The surface roughness increases with increase of exposure time and discharge powers. Sinha and Panigrahi [40] treated jute fiber with argon cold plasma for different time periods (maximum upto 15 min) and then fabricated composites with unsaturated polyester resin. Hydrophobicity of jute fibres was developed as a result of plasma treatment, as seen in scanning electron microscopy (SEM) photographs of treated jute fibre. They claimed that the hydrophobicity was a cause of the influence of oxidation on the fundamental component of fibre following plasma treatment. Kafi et al. [41] employed helium plasma to modify the surface of jute fiber. The resulting biocomposites developed from plasma treated jute fiber showed an increase in flexural properties and inter laminar shear strength compared to that of untreated jute composite. The process including plasma activation and acetylation, graft polymerization with vinyl monomers, which are very well described in the above literature review, improved the roughness of jute surface, but increased the cost of fiber.

1.5 Spectroscopic Analysis of Fibre Surfaces and Interfacial-Interaction

1.5.1 Infrared Spectroscopy

Due to its simplicity of use and abundance of data, FTIR spectroscopy is by far the method that is most frequently utilised. Alternate modes enable the use of various sample geometries, providing researchers with access to somewhat different facets of the chemistry to be studied (for instance, varying incident beam depths into the samples) or merely enabling them to choose the best sample preparation by choosing the appropriate FTIR mode to be used (transmission, attenuated or diffuse reflectance, among others). FTIR has been used to examine various fibre types and their functional group structures. The surface alterations upon the fibres have been investigated extensively, but the effect of this modification as a whole on the composite is a distinct aspect.

The first study to show an interfacial response between fibres and a polymeric matrix was conducted by Zadorecki and Flodin [42]. They created two coupling agents based on trichloro-s-triazine and modified it with varying terminal unsaturated groups to increase the contact between cellulose fibres and an unsaturated polyester matrix. They showed a covalent relation between the coupling agent and cellulose of the fibres and the copolymerization of the treated fibres with styrene utilizing FTIR and XPS (x-ray photoelectron spectroscopy) methodologies. Behera et al. [43] examined the FTIR spectra of raw jute and displayed the results in Fig. 6, where the strength of the broad, recognizable peak for -OH extending at 3200–3500 cm^{-1} diminished following alkali treatment. After alkali treatment, the peak's intensity decreased because the hydrogen bonds between cellulose and hemicellulose were broken. In raw jute fibre, an ester linkage between the carboxyl groups of hemicellulose and the hydroxyl groups of lignin was observed. This linkage was characterized by a prominent peak at about 1750 cm^{-1}. The ester connection between the hydroxyl

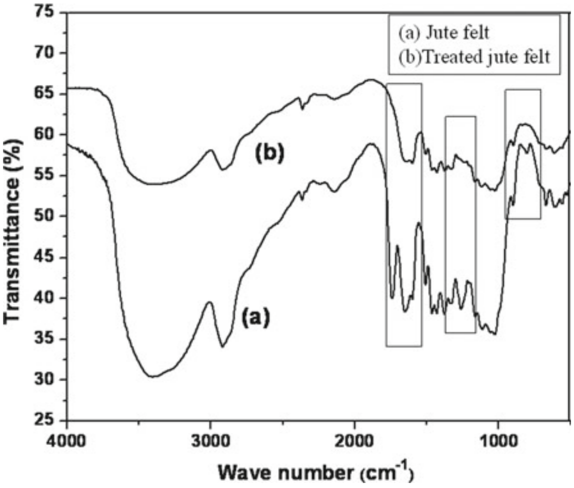

Fig. 6 FTIR spectroscopy of **a** raw jute fiber, and **b** alkali treated jute fiber

groups of lignin and the carboxyl groups of hemicellulose in alkali-treated jute fibres was broken, which caused the peak at 1750 cm^{-1} to vanish. Furthermore, the peak for the C-H stretching vibration in hemicellulose dropped (to 2910 cm^{-1}) after alkali treatment, indicating that the hemicellulose was removed. The connection between natural fibre and functional groups of the soy matrix was enhanced by this change in outcome.

When wheat straw was subjected to enzymatic treatment, [44] observed two distinct peaks at 2916 and 2850 cm^{-1} in the FTIR spectra of the straw's outer surfaces. These peaks are attributable to the asymmetric and symmetric stretching of -CH, which is mostly present in plant waxes. However, these peaks begin to almost disappear as soon as the enzyme started working, which helps to explain why lignin and polysaccharide lose some of their -CH2 content. A fascinating DRIFT spectroscopic analysis of composites made from bleached eucalyptus kraft pulp with polyethyleneimine was published by De la Orden et al. [45] from a similar angle (PEI). A band at 1715 cm^{-1} (C = O stretching vibration) was observed for carbonyl and carboxyl groups due to the oxidation of cellulose at high temperature.

Using a methacrylic anhydride glue, [46] worked with polystyrene and wood flour, studying their covalent bonding with FTIR. The peak at 1657 cm^{-1} in the spectra of the grafted wood flour indicates that the anhydride's related unsaturation resulted in a weak bond. This band greatly decreased when combined with styrene, and because of the phenyl group in styrene, modest absorption peaks appeared between 700 and 760 cm^{-1}. The composites' ^{13}C-NMR data verified the same.

Lu et al. [47] investigated the interface in the presence of two different coupling agents: maleated polyethylene (MAPE) and malleated polypropylene in composites made of wood fibre and high-density polyethylene (HDPE) (MAPP). The created composites were extracted with hot xylene using the Soxhlet method, dried, and then subjected to a transmission FTIR analysis. The most intriguing features in the FTIR spectra of the unextractable composite materials are the peaks that arise in the ester carbonyl area and the significantly increased absorption bands in the C-H region (2900 cm^{-1}).

1.5.2 Raman Spectroscopy

The amount of monochromatic radiation that scatters when a sample is exposed to high-intensity monochromatic radiation, such as that from solid lasers, depends on the chemical structure of the molecules being irradiated. Despite the fact that the peaks corresponding to the vibrational modes active in Raman and IR spectroscopies have the same energy shifts or occur at the same wavenumbers. However, the variances are also significant because there are differences in the relative peak intensities and the potential for some peaks to be absent under certain conditions. Raman is suitable for water samples, unlike infrared spectroscopy, where water significantly interferes. The lasers also stay away from the cellulose's and its derivatives' fluorescence. Jute fibre with carbon nanotube (CNT) adherence and its workability were the focus of [48]'s research. To investigate the non-covalent adhesive forces, bind the two together, they used Raman spectroscopy. The hemicellulose structure and the presence of aromatic benzene rings make CNTs and jute fibres both Raman active materials. The spectra contain three distinct bands at around 1340, 1575, and 1610 cm^{-1} that are the CNT tangential modes. Peaks were changed to 1340 to 1352 cm^{-1} when this was contrasted with jute-CNT, though. These alterations show that there is strong interfacial adhesion between the two.

Raman spectroscopy of Luffa fibres treated with carbonates and calcium phosphates was investigated by Parida et al. [49]. Using this method, they were able to discriminate between crystalline and amorphous cellulose and draw comparisons between this modified substance and hydroxy apatite, a mineral that makes up the bones and teeth of living things and is chemically comparable to it. As a result, it opens the door for more effective usage of the substance in biocomposites.

1.5.3 Solid-State Nuclear Magnetic Resonance (NMR)

By comparing sugarcane, coconut, and sisal fibres using cross-polarization magic angle spinning (CPMAS) ^{13}C-solid state NMR, early researchers like [50] had studied on the very basic property of the fibres, i.e., strength, and explored the packaging of molecular chains. Each type of fibre had a distinct peak for the cellulose that was both amorphous and crystalline, which was typically between 65 and 105 ppm. It is evident from comparing the CPMAS ^{13}C NMR spectra of different fibres that even if their fundamental structures are the same, each fibre has a different spectrum due to chain packing and molecular dynamics. It was shown that while these fibres can be homogeneous, they are typically amorphous by comparing the ^{13}C CPMAS with dipolar dephasing (CPMASDD) spectra obtained for the various analyzed fibres to those discovered by the CPMAS technique. An understanding of the packaging of fibrils is provided by smaller chemical shift values. This explains how each fibre, despite having the same fundamental structure, has a different installation.

Other works have described the relative locations of lignin, hemicellulose, and cellulose, the three main constituents of natural fibres, with lignin's aromatic carbon rings being visible at concentrations between 160 and 110 ppm. The presence of acetyl groups in hemicellulose is also confirmed by peaks at 21 ppm for methyl and 172 ppm for carbonyls. According to [51], the cellulose anhydro glucose units were represented by a single peak at 105 ppm. When working with the raw fibres, we can access these peaks. These will differ from surface-modified fibres, and matrix mixed composites will unquestionably exhibit various adhesive bonding as well.

1.5.4 X-ray Photoelectron Spectroscopy (XPS)

Another spectroscopic technique for investigation is XPS, which is applied to comprehend the integrity and efficacy of the interfacial bond. For chemical analyses and comments on the material's chemical make-up, it makes use of the photoelectrons that the material's surface emits. XPS is used to investigate the surface modification of cellulose fibres brought on by chemisorption with stearic acid. Studies utilizing XPS on cellulose showed that when samples were isolated using organic solvents, the ratio of oxygen to carbon atoms on the surface significantly increased.

The cellulose surface treated with triazine coupling agents was examined by Zadorecki and Ronnhult [52] using the XPS technique. This revealed that the triazines were not equally dispersed throughout the fibre but were primarily located on the surface. It can be assumed that the two main species in lignocellulosic materials are oxygen and carbon, and that the existence of minor elements like calcium, silicon, etc. will have an impact on how the interface develops. Additionally, the surface-treated fibres exhibited shifts in the O and C peak locations, similar to silanes and isocyanates. This bolsters the argument that surface treatment also influences bonding type and, consequently, interfacial adhesion.

According to Sgriccia et al. [53], the XPS data showed that kenaf was more hydrophilic than hemp. The increased oxygen-to-carbon ratio shows hydrophilic nature of natural fibre. The main constituents of plant fibres, including cellulose, hemicellulose, and pectin, have an O/C ratio of 0.85; lignin and pectin, on the other hand, have an O/C ratio of 0.35. As a result, it was determined that the fibres used by Sgricca had a higher concentration of waxy compounds when they were left untreated. This aids in our understanding of how the interacting boundaries between the matrix and fibres in a natural fibre composite develop over time.

The XPS data functioned as a good confirmation test about the removal of the primary cell wall of the natural fibres following chemical and other surface treatments, according to [54] 's study. They looked at the influence of enzymatic treatment on hemp and flax fibre and found that it had a beneficial effect because the post-treatment lignin and pectin concentrations decreased. The O/C ratio of 0.71 in polygalacturonase-treated hemp fibres indicates that the cellulose bundles are now revealed as a result of the removal of the outer non-cellulosic components. However, after xylanase treatment, there were few changes in the O/C ratio, indicating that the cellulose bundles were unharmed.

Through XPS experiments [34], the impact of corona treatment on miscanthus fibres was examined, and the change of peaks related to various C and O bonding were thoroughly investigated. After the corona discharge was applied to it, surface peaks like C = O, O-C = O, C-OH, etc. were increased while peaks connected to C-C and C-H bonds decreased. A control was matched with each of the peak's matching values. Belgacem and Gandini [55] have also proposed that this was caused by conceivable interactions between carbon and oxygen atoms on the fibres' surface and in the atmosphere.

2 Conclusion and Future Perspective

Natural fibres have been discovered to be more appealing than the traditional reinforcing synthetic fibres for entirely biodegradable composite production. Strength and aesthetic qualities are provided by natural fibres used as a reinforcing agent in composite materials. In the current study, the effectiveness of various surface treatments on natural fibers including alkali, silane, plasma, gamma, and enzyme treatments to improve interface interaction in corresponding polymer composites was the focus of the study. In order to investigate natural fibre reinforced polymer composites, FTIR, Raman, NMR, and XPS were used. These composites demonstrated enhanced physical and mechanical properties as a result of a better fibre resin interface. An efficient combination of treatments and the selection of natural fibre as a predecessor drive structural research toward the creation of biocomposites-based goods for the market. In order to accurately anticipate the product lifespan, we need to be able to predict the durability of the natural fibre composite using a variety of ways.

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Effect of Interfacial Bonding Characteristics on the Tensile Properties of Kenaf Fiber Reinforced Composites

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Abstract Natural fibers are widely used owing to their vicinity friendly. Amongst all the natural fibers, the utilization of kenaf is more beneficial because of its huge availability, less cost, withstanding ability for adverse climatic conditions, and fast growth. In the case of considering the kenaf fiber's capability of mechanical strength which depends on the matrix material like all other natural fibers. Herein, the focus is on given the interfacial bonding of the kenaf fiber at various combinations. The secondary consideration is given the tensile characteristics of kenaf fiber-reinforced composites.

Keywords Kenaf fiber · Interfacial bonding · Tensile property · Bond strength

1 Introduction

Fibers from kenaf plants, cotton plants, and okra plants are commercially available and inexpensive especially in contrast to some varieties of natural fibres [1, 2]. Use of kenaf fibre has advantages over other natural fibres because of its fast expansion, inexpensive, wide accessibility, and global weather tolerance. As a result of its rapid rate of carbon dioxide accumulation, kenaf as well has the greatest negative impact on the environment [3, 4]. The annual herbaceous plant with a mean diameter of 67.6 nm [5] has a name with Persian origins because of its short days and warm seasons. Growing kenaf requires very little in the way of pesticides because the plant is so sturdy and resilient thanks to its fibrous stalk [6]. The cultivation of kenaf for its fiber [7]. Although kenaf fiber is comparable to synthetic fibers in terms of thermal

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resistance, it is still inferior to natural fibres. Grown from seed in just three months, kenaf can reach a height of over three metres as well as a base circumference of three to five centimetres in a variety of climates [2]. Kenaf can flourish in a variety of soils with only minimal chemical treatment, typically consisting of some fertiliser and a single treatment with a herbicide [6]. The kenaf plant is made up of three different kinds of fibre: the bast, the core, and the pith [8]. Kenaf, on the other hand, has two types of fibers—the bast and the core—which account for 35 and 65%, in both [9]. It is possible to tell the bark from the core fibre based on their chemical make-up and structural differences [8]. All the cells in the pith are parenchymatous, and they're all polygonal rather than irregularly shaped [7, 10–12].

Natural kenaf fibre has a lot of potential compared to other fibres. Researchers have discovered that kenaf fibres, unlike glass fibres, are not abrasive during processing [3], and that they have suitable vibratory and physiological characteristics for use as reinforcement in polymer composites [13–16]. Kenaf fibres have been measured to have tensile strengths of 11.90 and moduli of 60.10 GPa [17], respectively. This is a simplified version of the plant cell wall as it appears in nature. Microfibril, microfiber, and primary/elementary fibre are all common names for this structure [17, 18]. The physiological and structural characteristics of kenaf fibre reinforced polymer composites vary with the fibre length, fibre content, and fibre orientation [19].

Researching the cellular ultrastructure and fibre morphology of kenaf can be done with great success using transmission electron microscopy (TEM) [9]. There is a wide range in the magnitude, form, and framework of the cell membrane fibres found in cross–sectional areas of kenaf [7, 9]. The cell wall fibres are made up of inter–cellular strands (foremost and supplementary wall–S1, S2, and S3) as well as its core fibres. Researchers have found that different types of kenaf fibres have different densities, tensile strengths, tensile moduli, and elongations at breaks depending on their primary soaking technique, country of source, and remaining parameters [5]. Among the many engineering applications for kenaf fibre, one of the most common is as a reinforcement fibre in polymeric materials [5, 7]. There are several uses for the fibres and leaves of the kenaf plant [20]. Forty to sixty percent cellulose, five to twenty percent lignin, and as much as twenty percent water are found in kenaf fibres, on average [21]. According to the study authors, the chemical makeup of kenaf fibres determines the fibres' overall properties [18]. Fibers' durability and rigidity come from cellulose's structural components, which do so via hydrogen atoms and other linkages. The fibres' hemicellulose content allows them to absorb water, decompose biologically, and break down when exposed to high temperatures. UV light breaks down the fibres, and the thermally stable lignin is to blame [17].

The potential for using kenaf plant parts in aesthetically related locomotive components like the dashboard, the floor, and the accessories was recently reviewed [20]. Kenaf is used to make the door casings, bumper, and footrest [22–26]. Among the many notable properties of kenaf fibres are their low pesticide and weedkiller requirements, low feedstock costs, and high biomass content. Crop rotation that includes kenaf fibre has many advantages, including weed control, drought resistance, increased biodiversity on solid ground, and the prevention of soya bean roundworm infestations. There is a need to be value added to kenaf fibre for it to be a viable alternative crop. Over the past few centuries, kenaf has been primarily utilised for its ability to be woven into cords, axing, and cloth. Lately, it has been utilized as a natural resource replacement for timber in the pulp manufacturing, helping for reduction of deforestation. In the textile and auto industries, it has been used to create nonwoven mats [17]. Opportunity to substitute an artificial fiber elements like carbon and glass with kenaf fibre for a broad scope of implementations [27, 28] due to kenaf's outstanding impact strength and high aspect ratio when was using as reassurance in polymer composites.

2 Whole Stem Fiber

Melt blending was used to create and characterise the composite material of whole stem kenaf fibre (WSK) and Ecoflex (compostable thermoplastic). Different fibre loadings and soaking times in NaOH solutions of varying strengths were used to prepare the composites. Using a Brabender internal mixer heated to 130 degrees Celsius for 10 min, composites with varying amounts of fibre (10, 20, 30, 40 and 50%) were compounded. In order to create biodegradable kenaf/Ecoflex sheets, the composites were pressed using compression moulding. Mechanical properties were tested before and after loading with kenaf fibres and after treating the surface of WSK fibres with NaOH. When compared to untreated WSK fibre, the mechanical and bending properties were found to be improved by 40% fibre loading, and by 4% NaOH in the treated WSK fibre. The FTIR analysis confirmed that the hemicellulose and lignin on the surface of the WSK kenaf fibres were removed by the alkali treatment. NaOH treatment's impact on composites' tensile strength, based on a 40% fibre loading. The tensile strength was found to be 16.5 MPa at a concentration of 4% NaOH and 9.2 MPa at a concentration of 2% NaOH. Lower NaOH concentrations were ineffective at cleaning the fiber's surface, leading to weaker fiber-matrix adhesion [29].

Effects of alkali treatment of kenaf fibre by varying absorptions of sodium hydroxide were investigated by Edeerozey et al. Analysis using scanning electron microscopy revealed that fibre treated with 3% NaOH failed to remove surface impurities, while fibre treated with 9% NaOH displayed the cleanest surface. Edeerozey et al. [30] found that WSK allowed to treat with 4% NaOH increased tensile strength by enhancing interfacial bonding and creating more sites of adhesion strength, thereby facilitating increased resin/fiber interconnection at the surface. Additionally, by removing both natural and artificial impurities, the alkaline treatment enhances the adhesion characteristics of the fibre surface by creating a rough topography. Alkali treatment has been shown to cause fibrillation, the separation of fibres within a bundle, which surges the operative area in interaction through the medium. There was a decline in tensile strength at higher concentrations because lignocelluloses degraded and fibre surface ruptured [31–33].

Modification of the composite's tensile modulus due to NaOH treatment, with a 40% fibre loading. After being subjected to a high enough concentration of NaOH, the fibre's tensile modulus value increased. Shipment allocation among the fibre and matrices happens via the robust fiber/matrix boundary, so the yield of a well-bonded composite is a direct result of this [30]. Chemically treated composites have a greater tensile modulus than their untreated counterparts. That is because there is a robust interaction among the treated fibre and matrix, as evidenced by the experiment. Successful preparation and characterization of compostable composites predicated on 40% fibre loading of treated fibre. Soaking with chemical solution enhanced the fibre and matrices contact by removing organic and hetero polymer, leading to good combination of fibre matrices [34], which in turn increased the tensile and flexural strengths of the composite.

3 Chemical Treatment

In order to strengthen the interfacial bond between the fibres and the polymer matrix, chemical surface modification is a common technique. The vibratory and physiological characteristics of kenaf fibre may change as a result of its alkaline treatment, which may alter not only the fiber's surface but also its texture. The purpose of this research was to determine the changes in fibre properties that occurred under varying alkaline treatment conditions, specifically alkali solution concentration and immersion time. In total, 360 fibre samples were tested under 24 unique combinations of starting conditions and alkaline exterior alterations. Finally, three methods were used to analyze and report the results: regression, data averaging, and compliance management system. Furthermore, an electron microscope was utilized for examining the test samples of visual characteristics, rupture zone, and fiber size. As can be seen from the results, unprocessed kenaf fiber has a mean diameter of 68 m, a thickness of 1.2 g/cm^3, and a resistance to the tension of 780 MPa. In addition, 5% sodium hydroxide was superior to treating kenaf fiber due to not placing any stress on the fiber's surface and construction, unlike the 10 and 15 percentage of sodium hydroxide.

Scanning electron microscopy (SEM) was utilized to investigate the fiber surface morphology. The chemical surface treatment results in a hygienic and harsh exterior on the fiber, as shown in Fig. 1, which is crucial for the interlayer adhesion of polymer and kenaf fiber. Unprocessed fiber has lignin, hemicellulose, and wax on its surface, all of which can be removed with a chemical process. As a result of the treatment, the surface of the fiber is not perfectly smooth, leading to improved interlocking between the polymer and the fiber. Kenaf fiber has the right characteristics for engineering application in the FRP industry. The use of chloride-containing clean water in the first stage of kenaf fiber's water purification may cause structural damage to the fiber and a decline in the fiber's mechanical properties. Therefore, the water retting process should not be performed using chlorinated drinking water. There was no discernible difference in the elastic modulus of kenaf fiber regardless of the location of initial treatment (natural body of water, synthetic lake with natural water, or river).

Damage to the fiber's texture occurred during alkaline treatment with 10% and 15% NaOH solution, as the preserved fiber is extra warped, significantly better, and so fragile than the elevated fiber. Therefore, alkali treatment with such high levels of NaOH solution is not advised. For kenaf fibers, one could estimate a mean diameter of 67.6 m. The results of using the regression, data averaging, and compliance management system to analyze the data show that the results are very close. As the NaOH concentration of the solution and time of immersion increased, the young's modulus of the kenaf fibre decreased. The alkaline treatment did not significantly affect the tensile properties of kenaf fibre across groups, but it did affect the maximum strain values [5].

4 Woven Kenaf Fiber

Organic fibre reinforced polymer composites have been explored for their potential to benefit from hybridization in addressing the issues of enhanced humidity acceptance, imbalanced vibratory characteristics. Hydrophobic polyethylene terephthalate (PET) fibre was employed in the fabrication of woven kenaf cemented polyoxymethylene due of its strength and versatility (POM). The resistance to tension of the woven POM/kenaf/PET hybrid composite rose from 72 to 85 MPa in tests conducted along the kenaf fibre direction. The interwoven hybrid composite of POM, kenaf, and PET saw a rise in its tensile modulus from 67 to 75 MPa. Bending resilience for the hybrid POM/kenaf/PET composite was 160.1 MPa, whereas for the woven POM/kenaf composite it was 90.3 MPa, and bending strength for both composites was 191.4 MPa. As a bonus, the hybrid interwoven composite had much greater toughness than the twisted POM/kenaf composite. The combination with PET fibre significantly decreased the humidity acceptance of weaved POM/kenaf composite around 30%. The results [35] show that the woven composite's ductility, implications, and rigidity with respect to moisture absorption were all greatly improved through hybridization with PET fibre (Fig. 2).

All the woven composites' tensile and modulus readings were taken along the kenaf fibres' axis. Intricately intertwined kenaf/PET hybrid composite has a much greater tensile strength than woven POM/kenaf composite or interwoven POM/kenaf/PET hybrid composite. Matrix formation could have been caused by weaving kenaf and PET yarn together. Incorporating mechanical bond of the reinforcements into the composite' process of production improved their wettability. To top it all off, the weaved POM/kenaf (70/30) composite has a tensile strength roughly 19% higher than pure POM, at 75 MPa compared to 61 MPa. Synthetic morphology of a woven POM/kenaf composite. Micrographs show that the kenaf and POM connect very strongly at their interface, which is what gives the composite its increased tensile strength above pure POM. Fiber rupture or matrix cracking are the most common

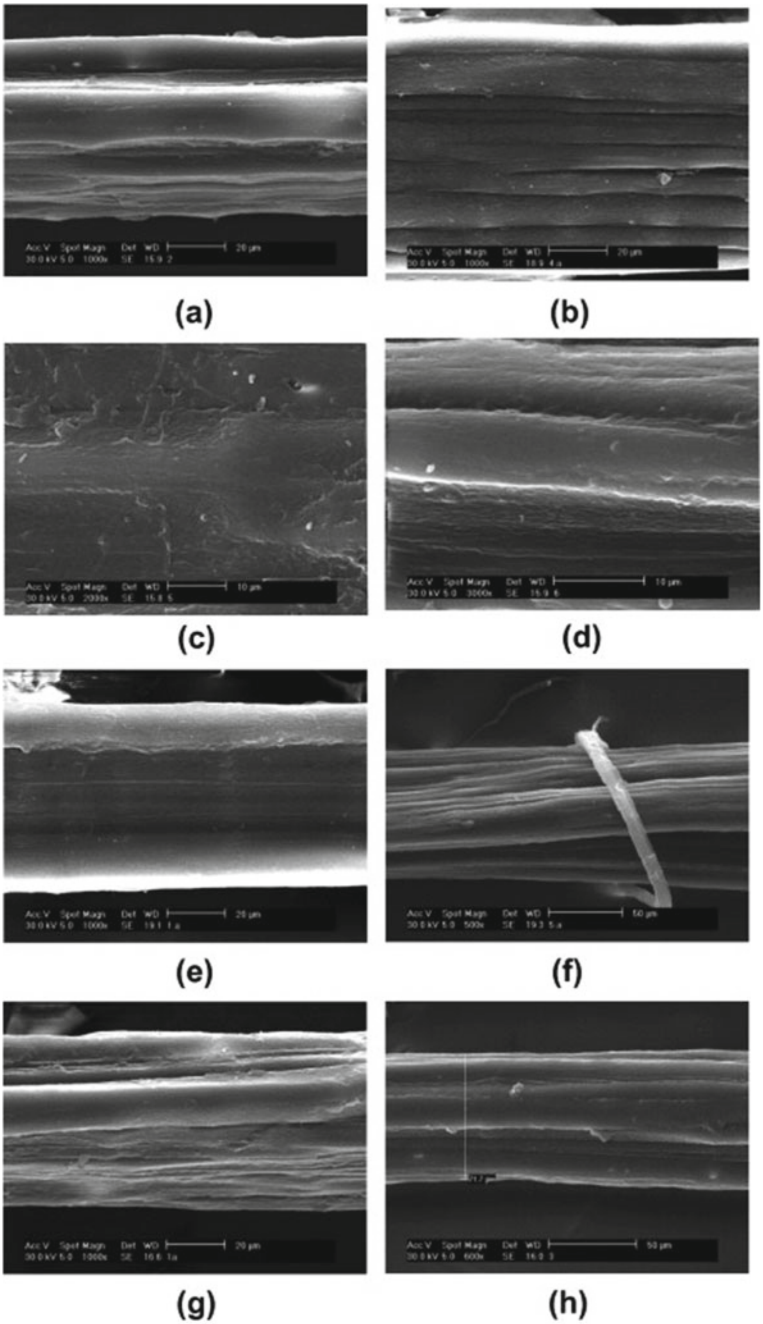

Fig. 1 SEM images kenaf fiber untreated **a, c, e, g** and treated **b, d, f, h** [5]

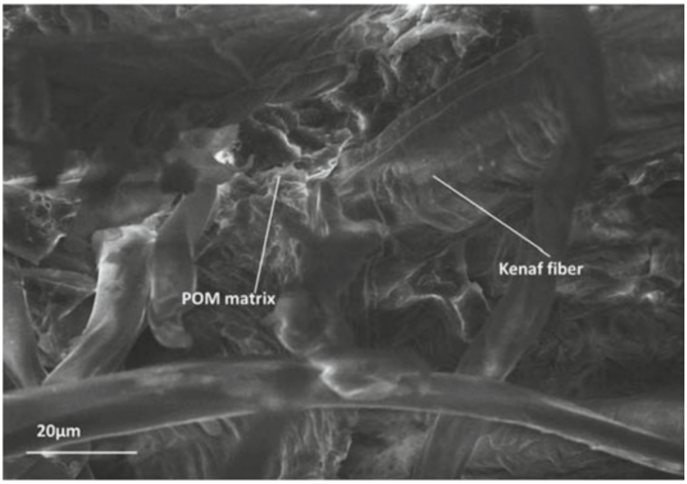

Fig. 2 FESEM images kenaf fiber POM matrix interfacial bonding [35]

causes of damage in woven composites. Fibre pullout is quite improbable because of the tight weave of the reinforcements and the matrix's resilience to it [36, 37].

The elastic properties of the interlaced POM/kenaf/PET (70/15/15) composite material are roughly 40% higher, from 2.3 to 3.8 GPa, than that of the woven POM/kenaf (70/30) composite. Based on the results, the woven POM/kenaf/PET composites has a greater elastic modulus than the interlaced reinforced hybrid by almost 10%, from 3.8 to 4.2 GPa. It is possible that the high elastic modulus of the POM/kenaf/PET hybrid composite was caused by the weaving process. In addition, the results demonstrated that an increase in the reinforcing weight percentage greatly improved the formulations' tensile and elastic moduli. The effects of PET fibre orientation on the elasticity and tension of woven composites. The tensile strength measured perpendicular to the kenaf fibres was 89 MPa, which is more than the 85 MPa recorded in the orientation of the kenaf fibres. The elastic modulus produced for the POM/kenaf/PET (70/15/15) composite material was about 13% lower, at 3.3 GPa, than that obtained along the kenaf fibre direction. Results like these suggest that the PET fibre was mostly responsible for increasing the composite's tensile strength, whereas the kenaf fibre was primarily responsible for increasing the composite's elastic modulus.

Strong tensile power was measured in a FESEM image of the interfacial adhesion among PET fibre and POM. If the fibre and matrix are going to connect well at their interface, the fiber's surface tension must be higher than the matrix's. Strong adhesion is shown by the coupling among POM/kenaf and POM/PET, and this can be explained by the matrix efficiently wetting the fibres in both situations. Possibly this is due the matrices can break through the mechanically interlocked fibres of the kenaf/PET fabric. Poor contact interaction with polymeric matrix has been linked to the low mechanical characteristics of alkaline-treated natural fibres [38].

The interlaced hybrids composites' qualities, as determined by mechanical testing, are superior to those of the woven POM/kenaf composite owing to the matrix's-controlled pressure dispersion. The tensile strength of the hybrid interwoven material, as evaluated in the orientation of the natural fibres, increased from 72 to 85 MPa when the fibre content was increased. The tensile strength of the woven POM/kenaf composite also increased, from 67 to 75 MPa. Compared to the woven POM/kenaf composite, whose bending resilience declined from 191.4 to 90.3 MPa, the hybrid POM/kenaf/PET composite's resilience was reduced from 160.1 to 104.9 MPa. The combination fabric's compact construction was a result of the high adhesive strength of the kenaf fibre and the PET fibre, which prevented fibre pull-out throughout the warp yarns. The test findings showed that the impact strength also rose considerably as a response of the regular stress dispersion in the matrix. As a bonus, the hybrid composite outperformed the POM/kenaf composite in its resistance to water absorption. According to the study's findings, including PET fibre into the composite improves its ductility, consequences, and resistance to water absorption [35].

5 Kenaf With Polyethylene

It was investigated how adding a compatibilizer to a kenaf fibre strengthened, high-concentration polypropylene blend would affect the material's mechanical qualities. When homopolymer hydroxide transplanted high concentration polypropylene was used as a coupling agent in the composites, improvements were seen in both the materials' vibratory properties and their adhesion behaviour. A more harmonious relationship among the matrix and fiber is responsible for these improvements. The 8% MA-HDPE loading was the most beneficial in terms of ductile and bending qualities when compared to the other compatibilizer contents. However, the interlayer nature and vibratory characteristics of un-compatibilized composites were shown to be subpar in comparison to those of compatibilized composites. Fourier transformed infrared spectroscopy corroborated the structural modifications, revealing a bending vibrations of ester carbonyl groups ($C = O$) among both 1725 and 1742 cm1 and a maximum of hydroxyl group at 3327 cm1. This occurs because maleic anhydride forms a strong interfacial interaction with the kenaf fibre during the esterification step. Examination of a tensile fracture's surface morphology with FESEM demonstrated improved included action [39].

Tensile properties are those of a material that change as a result of being subjected to tensional forces. The tensile modulus, strength, and strain that result from applying that force are the main topics of discussion here. Impact of compatibilizer on ultimate strength and elastic modulus of composites at 8.5 and 17.5 wt% fibre loadings. Higher fibre loading composites (17.5 wt.%) showed increased tensile strength, especially when compared to those with a lower fibre loading (8.5 wt.%). The tensile strength of composites increased after the fibre fraction reached a threshold value [40]. With a higher fiber-to-matrix weight ratio, more of the applied force is transferred to the fibres, leading to greater tensile strength [41]. Every compatibilized composite outperformed its non-compatibilized counterpart in tensile strength. Hydrophobic polymer matrices and hydrophilic fibres may be incompatible, which contributes to the low tensile strength of unstabilized composites [42]. From the micrograph of the fracture surface under tensile stress, the un-compatibilized composites with the 8.5 wt% fiber content are failing. Plastic deformation of the host matrix occurs during tensile testing, leading to a porous and uneven surface. At a fiber loading of 17.5 wt.%, additional holes were observed, though the matrix texture was already present in irregular forms. More so, the matrix did not adhere particularly well to the fibers' outer sides. Spaces or cavities between the fiber and matrix are often signs of insufficient adhesion at the interface. Without a compatibilizer, micrographs of composites show that the fiber is easily broken and pulled out due to a lack of cohesion between the monomer and the fiber [42–45].

Tensile strength of the composites rose by 7.4, 14.5, and 15.9% when a compatibilizer of 4, 8, or 12% was added, respectively, in comparison to the un-compatibilized composite (20.42 MPa) at a fiber loading of 8.5 wt.%. The composites' increased tensile strength following MA-HDPE incorporation is proof that the kenaf fiber and HDPE have formed a solid bond. More effective stress transmission from the matrix to the fiber was achieved in the addition of a compatibilizer because of stronger interfacial adhesion between the fibre and the matrix [46–50]. To improve the composite's strength, it is suggested that the anhydride groups of MA-PP be esterified with the hydroxyl groups of cellulosic fibers [51]. An intensive emulsification and polymer backbone hydrogen bonding reaction was seen, as was validated by FTIR analysis, indicating that the compatibilized reinforced scheme was indeed successful. Changes in the shapes of the substances were brought about by the compatibilizer. The use of MA-HDPE as a compatibilizer significantly improves the interfacial adhesion between the fibre and the matrix, as seen by the cracked surface of compatibilized composites. Clearly visible in the micrograph is the tight interfacial adhesion and high water sorption of the fibres in the matrix, attesting to the strength of the connections between the two components.

After a tensile test fails, at any compatibilizer concentration, the fibres are still attached to or in touch with the matrix, showing a high degree of polymer coverage on the pulled-out fibres and strong adherence to the host matrix. The fibres were found to adhere strongly to the polymers, as evidenced by the fact that they were dipped and extracted together with the bulk matrix. The tensile strength of the composite at a fibre loading of 17.5 wt was improved by increasing the compatibilizer loading from 2 to 8%. The compatibilizer resulted in a 13.6% increase in tensile strength over unstabilized composites (22.15 MPa). Adding a compatibilizer of 12% to a fibre loading of 17.5 wt.% reduces tensile strength somewhat. Most fibres, as described by few experimenters [52] up until the saturation stage of the compatibilizer, are in the interphase between the combination and the matrix material. The homogeneity and, by implication, the vibratory characteristics of the mixes are altered when the compatibilizer percentage is raised above the saturation level, as only a percentage of the particles stay in the contact region and the rest are diffused in the matrix. It was evident that the fibre had integrated into the HDPE matrix material when MA-HDPE was introduced at concentrations of 8 and 12%, and that there were no gaps among the fibres and the polymer matrices.

The fact that fibres are fracturing under experimental conditions is also indicative of enhanced interfacial adhesion. Successful adhesion may be seen in each of these cases. This reveals that the incorporation of MA-HDPE enhanced the fibre's interfacial adherence to the matrix. Thus, it is clear why compatibilized composites outperform their non-compatibilized counterparts in ductile strength and modulus. It has been observed that the inclusion of the coupling agent in the composites system promotes the bonding between the fibre and matrix [53, 54]. This is because the anhydride group in the coupling agent strongly attaches to the -OH on the membrane of the natural fibre. The physical and hydrophilic properties of kenaf fibre strengthened HDPE composites were studied, together with the effects of feeding with an MA-HDPE compatibilizer. In the absence of a compatibilizer, the composites constituted by the kenaf fibre and the HDPE matrix exhibited poor physical and interfacial properties. MA-HDPE was used as a compatibilizer to improve the tensile and flexural properties of kenaf fibre strengthened HDPE composites.

Fracture surface characterization reveals that the compatibilized materials are better uniformly distributed on the surface and have enhanced interfacial adhesion. Increased mechanical characteristics and matrix-to-kenaf fibre adhesion were observed with 8% MA-HDPE loading compared to other macromolecular species contents. It is hypothesised that the chemical bonding among the acetic hydroxide on the coupling agent and the hydroxyl units of kenaf fibre accounts for the harmonious coexistence of HDPE matrix and kenaf fibre. FTIR data also backed up claims that the composites' mechanical properties and interface characteristics were improved. On the FTIR spectrometer, the extending frequency of the carboxyl ($C = O$) group in the ester was observed between 1725 cm^1 and 1742 cm^{-1}, and the hydroxyl group was observed at 3327 cm1, showing that the maleic anhydride had bonded to the kenaf fibre via isomerization process. These results imply that incorporating MA-HDPE composites into kenaf fibre reinforced HDPE increased interface attraction among the kenaf fibre and HDPE matrix, resulting in enhanced dynamical effectiveness and interfaces properties [39, 55].

6 Fiber Loading

Mechanical (tensile, flexural, impact, hardness, and abrasion resistance) and thermal (thermogravimetric analysis) properties of Kenaf bast fibre reinforced thermoplastic polyurethane (TPU) composites as a function of fibre content. The composite was made using a melt-mixing technique and then compressed using a mould. At least four distinct fibre loadings (at 20, 30, 40, and 50% weight percent) were made. The highest tensile strength was observed at a fibre loading of 30%, whereas the modulus rose with fibre content and the strain worsened. As fibre loading was increased, bending stiffness and elasticity were also enhanced. Reduced impact strength was observed as fibre loading was increased. The addition of 30% fibre content increased

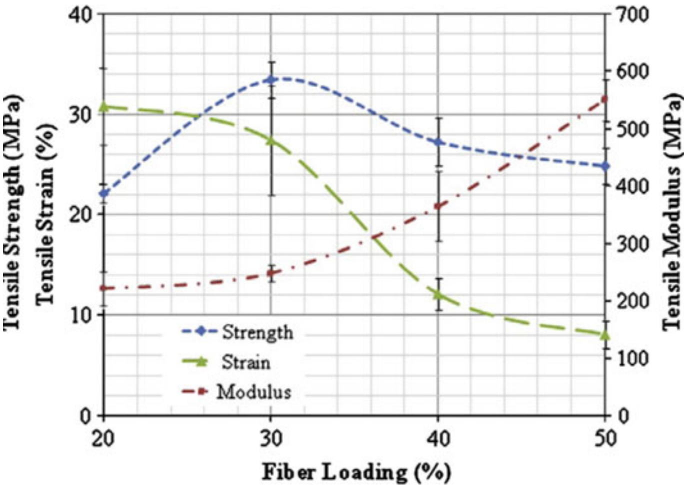

Fig. 3 Fiber loading-based tensile properties [55]

the hardness. The fibre loading has a negative effect on the abrasion resistance. The composite's thermal stability suffered as a result of the addition of fibres. TPU/KF composites' tensile characteristics change as a function of fibre loading, as seen in Fig. 3. Strength and modulus were lowest at 20% fibre loading, whereas strain was highest. For maximum durability, aim for a fibre content of 30%. The modulus decreases with increasing fibre loading, from 40 to 50%. The strength of composites depends on a number of variables, including the strengths of the fibres and matrix, the volume percentage of the fibres, and the fiber-matrix interfacial bonding. Matrix fibres serve as load bearers. Uniform and efficient stress distribution is more important for achieving good tensile strength [56].

One can see that the tensile strength drops dramatically with the amount of fibre loading. The incapacity of the fibres to share the burden is to blame for this phenomenon. So, with low fibre content, the impact of fracture initiation was more prominent than the impact of fracture suppression [57]. Maximum composite strength, 33.5 MPa, is achieved at a fibre loading of 30%. The improved bonding between the fibres and the matrix is responsible for this. There is an optimum level of fibre participation in stress transfer at a fibre content of about 50% [56]. The strength decreases by around 19 and 25% at the 40 and 50% loadings, respectively. Because the matrix is unable to adequately moisten the fibres at higher loadings, agglomeration occurs, and stress transfer is impeded [58]. The tensile modulus rose as the fibre loading increased. Since the composite's filler (the fibres) has a much higher stiffness than the matrix, it stands to reason that the composite's stiffness would grow as the volume of fibres was increased. For a given amount of fibre loaded, there was a decreasing strain pattern. This is another expected result of the low-strain expansion of fibre volume (the strain in kenaf is 1.6% [21]).

Variations in physical and thermodynamic properties of TPU/KF were observed across a range of fibre loading. Fibers were loaded at 20, 30, 40, and 50% of their weight in a series of experiments. Matrix/fiber interfacial adhesion was strongest at 30% fibre loading, indicating high tensile strength. The tensile modulus increased when the fibre loading was increased. [55] As fibre loading was increased, tensile strain tended to decrease.

7 Polyester With Kenaf

After incorporating kenaf fibre into unsaturated polyester resin at varying concentrations, the composites were cross-linked with methyl ethyl ketone peroxide and the activator cobalt octanoate. In order to increase the material's toughness, 3% liquid natural rubber (LNR) was included. To strengthen the adhesion of the kenaf fibres to the matrix, we soaked them in a sodium hydroxide solution. Impacts and bending testing were utilized to determine the composites' mechanical properties. Composites' ESCR (environmental stress cracking resistance) in both acidic and basic environments was also investigated. SEM/FTIR analysis and SEM imaging were used to evaluate bonding mechanisms. Researchers discovered that by using LNR, both the shock power and shatter hardness were improved. Improved shock and flexural strengths in the composites were attributed to the alkali treatment of the fibres. From an ESCR perspective, the composite in an acid media has the quickest permeation, following by the composite in a base medium, and finally the composite in the absence of medium [59]. This is shown in the SEM images presented in Fig. 4 [25].

8 Conclusion

The interface adhesion of kenaf fiber at different combinations is the primary focus of this article. The tensile properties of composites reinforced with kenaf fiber are secondary. The interfacial bonding strength of kenaf fibers in their various states is evaluated. Alkaline treatment of the stem fiber, taken as a whole, has been shown to increase its tensile strength. Chemical alkaline treatment increases tensile strength, but too much sodium hydroxide or too long of an immersion time reduces the benefit; therefore, it is important to determine the best alkaline treatment conditions for maximum tensile strength. Improved tensile strength is one of the many benefits of combining woven Kenaf with PET to create a composite material. Increased interface binding among the matrix and fiber in kenaf with polyethylene composite, in which the maleic anhydride has attached to the fiber via the esterification process, results in a notable increase in tensile strength. When it comes to fiber loading, 30% revealed the highest tensile strength, while increasing the fiber content raised the modulus and decreased the strain. Cobalt octanoate speeds up the cross-linking reaction of

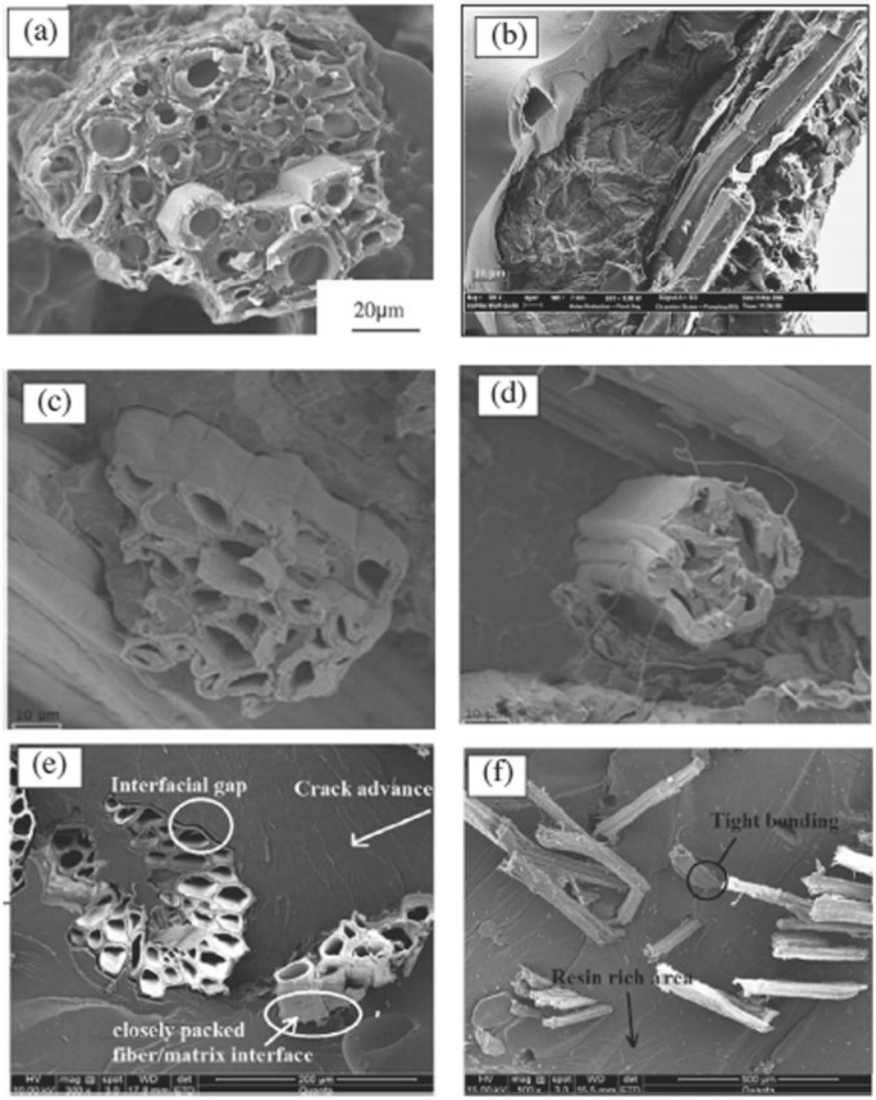

Fig. 4 SEM images of **a** kenaf fiber, **b–d** tensile fractured surfaces and **e** and **f** kenaf fiber composites [25]

an unsaturated polyester–kenaf composite, which has exhibited improved interfacial bonding with resin.

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Effect of Interfacial Bonding Characteristics of Flexural Fractured Pineapple Leaf Fibre Reinforced Composites

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Abstract It is the aim of this piece of research to investigate Pineapple fiber reinforced composites based on natural fibers that are both eco-friendly and possess unique properties, and to take into account their various properties and their eco-friendliness. As a result of their strength, lightness, and affordability, pineapple fibre materials are considered to be stronger, lighter, and more affordable than traditional materials due to the fact that they are stronger, lighter, and more affordable than natural materials. It has been an important research subject for scientists in recent years to investigate the use of natural fibres as reinforcement materials for polymeric composites that can be used in technical applications. There are many advantages of natural fibers, such as continuous supply, easy handling, and a biodegradable nature, which make them a good choice when it comes to textiles. Due to its low cost, low density, hardness, better tolerance for harsh weather conditions, and good performance thermally and mechanically, natural fibres have gained a great deal of popularity worldwide because of their low cost, low density, hardness, and environmental friendliness. A large number of crops are produced every year around the world, and most of the waste produced by these crops does not have any use whatsoever. It is estimated that thousands of tons of different crops are produced every

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year. As it is well known, agricultural wastes include leaves from pineapples, dates seeds, and shells from various types of dry fruits, as well as other organic wastes. As part of this project, we have combined the wastes from pineapple leaves (PALF) into a single product. There are numerous promising applications for metal, such as the use of metal as an alternative to the construction of automobile bodies in the future, which are currently being investigated.

Keywords Pineapple Leaf (PALF) · Epoxy resin · Mechanical properties · Structural strength

1 Introduction

During the last few years, polymeric composite materials have been gaining a lot of attention and benefiting from their use. As reinforcing materials, previous studies have focused on high performance polyamides, polyesters, aramids, glass fibers, and carbon fibres embedded within appropriate polymeric matrixes in order to achieve high performance reinforcement. In spite of this, these types of materials can be quite costly, as they are non-renewable and are in high demand. Since petroleum-based products and their prices are uncertain, there is a need to find natural alternatives to petroleum-based products [1]. The use of natural fibers that are available in abundance has been investigated for potential applications in consumer goods, furniture, and low-cost housing, such as sisal fibers, banana fibers, hemp fibers, jute fibers, palm fiber, kenaf fibers, jute fibers, bamboo fibers, coir fibers, henequen fibers, and pineapple fibers. As a matter of fact, the definition of a composite material is to be one that is a macroscopic combination of two or more distinct materials, embedded within the matrix phase by fibers, sheets, or particles that have been incorporated into it on a microscopic scale. There are several different types of reinforcing and matrix materials that can be used, such as metal, ceramic, and polymeric materials [2–5]. Composites are stronger and stiffer when fibers or particles are the primary load-bearing components in a composite rather than continuous matrix phases when they form the primary load-bearing component. In the event that the load is complex, the matrix can serve as a medium through which the load is transferred between fibers, or in the event that the load is complex, the matrix may serve as both a medium through which the load is transferred between fibers and matrices. A number of reasons have contributed to the increasing popularity of natural fibers across the globe, including their low cost, low density, hardness, durability, as well as their thermal and mechanical properties, as well as their help in caring for the environment. PALF, or pineapple leaf fibers, are used as textile materials in many countries that have been using them for a long time. However, despite the fact that they are mechanically excellent and environmentally sustainable, PALF fibers are the least studied natural fibers when it comes to incorporating them into composites, including as reinforcement [6, 7]. An extensive amount of research has been conducted on PALFs as well as PALF-reinforced composite materials. In this paper, we review techniques for extracting

PALF from lignin, characterizing the fiber, and applying PALF to different applications, modifying PALF, and discussing the properties of composites incorporating PALF as reinforcement. In order to maximize pineapple's "resource potential" and to energize PALF's use as an important reinforcement fiber in polymer composites, it is essential to develop value-added applications for PALF, such as using it as a reinforcement fiber [8].

In order to manufacture NFRC biocomposites from these post-harvest wastes, natural fibres can be extracted in micro and nano-sized particles which can be incorporated into the biocomposites. A number of advantages can be attributed to the use of natural fibres, and these include their cheap cost, biodegradability, abundant availability, clean processing, and no abrasiveness to cutting tools. There are many benefits that can be achieved by using natural fibres in the global agriculture market to increase food security and eliminate poverty. The production of synthetic fibres and polymers relies heavily on the use of conventional energy sources such as petrochemicals and crude oil [9–12]. There are more than 140 million tons of synthetic polymers produced every year, which is a significant amount. There are a wide range of applications for these products such as packaging, disposable consumer products, household-kitchen applications, the automotive field, the aviation field, and so on. When their use is over, these materials have to be disposed of in a way that does not harm the environment since they are not biodegradable. A total of approximately 1.5 Billion Tons of artificial fibers are produced every year, while there are approximately 2 Billion Tons of lignocellulose materials produced each year from crop waste. In addition to clothing, packaging, paper pulp, the automotive industry, building materials, and sports equipment, the natural fibres that come from this industry are used in a wide range of applications.

A great deal of crops are grown every year, but there is a great deal of waste generated from those crops. In addition to pineapple leaves and dried fruit shells, there are a number of agricultural wastes that can be recycled. It was found that graphene oxide nanopowder GO (NP) was one of the best materials for functionalizing pineapple leaf (PALF) and dates seed (DS). We are exploring the use of composite materials as an alternative to metals in the body building of automobiles. There is even the possibility of applying loads in a transverse direction to the direction of the fibers. Compared to fiber-based composites, composites are tougher due to the matrix's ductility, which makes them less brittle. When designed properly, a combination of materials will demonstrate greater strength than a combination of materials designed individually [13–18]. A variety of applications are found for composite materials, ranging from electrical applications to electro-thermal applications, environmental applications to tri-biological applications.

2 Materials and Methods

2.1 Materials

2.1.1 Pineapple Fiber

This study used a composite material that was made out of pineapple leaf fibres with a thickness of 0.3 mm as a synthetic reinforcement that was manufactured from pineapple leaf fibres. Using the fibres of pineapple leaves as a natural reinforcement, the structure was constructed using pineapple leaf fibres. It is the pineapple leaf fibres that are combined with epoxy resin in order to make a laminate, which is produced by combining pineapple leaf fibres with epoxy resin. We prepared the composites using two different methods as a result of the melting process and the solution mixing process we used as a result of the melting process. The effects of fibre orientation, as well as fibre length, fibre loading, and fibre orientation, were also investigated by researchers in addition to the effects of fibre length, fibre loading, and fibre orientation. Through the use of fibre distribution curves and fibre breakage during processing and in addition to analyzing the effects of fibre loading, fibre length, and modifications to the fibre surface on the properties of the composites, we investigated the optical and mechanical properties of PALF reinforced polyester composites. A linear relationship was observed between the modulus and the tensile strength of this thermoset composite as the content of fibre increased. There was also a similar pattern observed for the impact strength of the samples. The flexural strength of the material levels off when the fibre content exceeds 30% by weight. In composites reinforced with fibers that were treated, all mechanical properties were significantly improved as a result of the treatment [19]. There is, however, an increase in the storage modulus E’ when fiber loading is considered in dynamic mechanical thermal analysis as a result of fibre loading. Moreover, the chemical treatment was able to improve both the mechanical and thermal stability of the composition as well (Fig. 1 and Table 1).

2.2 Hand Layup Process

Hand laying composites is the easiest way to process them since they are the simplest to put together. In addition to this, there are not many infrastructure requirements associated with this method. There aren’t many steps involved in the processing of it. It is necessary to place thin plastic sheets at the top of the mold plate and at the bottom of the mold plate to ensure that high quality surface finish is achieved. This is done in order to remove the risk of the product sticking to the surface. Firstly, a release gel is sprayed on the surface of the mold so as to prevent the polymer from sticking to the surface and becoming clogged. A reinforcement material such as woven mats or chopped strands is laid after the Perspex sheet has been installed. The thermosetting

Fig. 1 Pineapple fibre

Table 1 Properties of Pineapple fibre

Properties	Value	Unit
Density	1.07	g/cm^3
Tensile strength	126	MPa
Young's modulus	4405	MPa
Elongation of break	2.205	%
Moisture regain	12	%

polymer will be poured onto the surface of the mat that has already been placed in the mold as soon as it has been thoroughly mixed with the hardener (curing agent) in liquid form [20–22]. Using a brush to spread the polymer uniformly, it is ensured that the polymer is uniformly distributed. When a roller with mild pressure is moved along the surface of the mat covering the polymer layer in order to remove any air or excess polymer present within this layer, the excess air or polymer is removed from the mat-polymer layer. It is important to spray the top mold plate with release gel when the plastic sheet is placed on the mold plate. Following the stacking of the top mold plate, there is an application of pressure to the mold plate. The natural pineapple fiber laminate that hangs over the edge of the mold can be easily trimmed off with a razor knife once the lay-up has gelled, but before it has hardened, after the lay-up has gelled but before it has hardened. Depending on the temperature, canalization, and the nature of the part, a curing process can take from 24 to 32 h, depending on the type of part. In the case of a female mold, the longer the cure time, the more shrinkage will occur and the easier it will be to part. There is a tendency for the part to come off easier when it comes from a male mold before it shrinks in an appreciable way (Fig. 2).

Fig. 2 Composite making process

2.3 Mechanical Testing

According to ASTM D -3039, composites prepared from Pineapple Leaf, Dates Seed, and Graphene Oxide Reinforced materials were evaluated through tensile testing according to the ASTM standard. In order to prepare the composite specimens, 250 mm long and 25 mm wide specimens with a thickness of 3 mm were prepared. There was a cross-head speed of 2 mm/min applied to the specimens, and they were tested until a failure was detected. For the purpose of measuring the flexural properties of the metal, three-point bend tests were conducted in accordance with ASTM D 790 M. There was a specimen that measured 125 mm in length, 12.7 mm in width, and 3 mm in thickness. In the bending test, the outer rollers were 64 mm apart and samples were tested at a strain rate of 0.2 mm per minute under the three-point bending condition. The three-point bend test was chosen for this project because it is more economical and reduces the amount of material required for each test as well as eliminates the need to use test equipment to determine the center point deflection accurately. In accordance with ASTM standard guidelines, a specimen is prepared according to its specifications. Impact tests are done in the Izod and Charpy labs in order to determine the impact strength. As per ASTM standard, a setup for impact has been created. There must be a specimen loaded in the testing machine and the pendulum must be allowed to swing until the specimen is fractured or breaks or both. It is possible to measure the impact strength of a material by using the energy needed to break it, as the impact test is used to note the energy needed to break the material.

3 Result and Discussion

3.1 Mechanical Strength

A tensile test, a flexural test, and an impact test, as prescribed by ASTM standards, are used to evaluate the strength of pineapple leaf-reinforced composite materials. A tensile test is performed according to ASTM D3039, a flexural test is conducted according to ASTM D790, and an impact test is performed according to ASTM D256, as per ASTM standards. As the pineapple leaf fiber reinforcement was increased to 15, 20, 25, 30, and 35% of the weight, there was an increase in the weight of the pineapple leaf fiber reinforcement. During the tensile test, dimensions will be 250 × 25 × 3 mm, flexural test dimensions will be 125 × 13 × 3 mm, and impact test dimensions will be 65 × 13 × 3 mm. These three tests will be performed in sequence: the tensile test, flexural test, and impact test. As a method of determining the mechanical strength of the materials, Kalpaks universal testing machines and Charpy impact testing machines were used. On neat-epoxy samples that have been prepared in accordance with ASTM standards, mechanical tests are first conducted using ASTM standards [23–26]. According to the results of the neat epoxy composite (10:1 ratios), the tensile strength of the resin compound was 12.34 MPa, the flexural strength was 21.42 MPa, and the impact strength was 0.10 J with the corresponding thickness of the composite. Various tensile strengths of composites reinforced with pineapple leaves were determined, ranging from 14.6 MPa to 29 MPa. There is a normal range between 32.12 and 46 MPa for the strength of the pineapple composite flexure. In general, pineapple composites exhibited improved impact properties and were able to reach a maximum impact strength of 0.4 J. With a high degree of mechanical strength relative to the other polymer composites, the 30% of pineapple leaf-reinforced composites exhibited improved impact properties.

There is a proper mixture between 30% of pineapple leaves and secondary matrix materials in this formulation. Unlike the other samples, this sample does not contain any voids; hence, it has a stronger strength when compared to them. The increased fiber weight percentage (35%), in combination with the agglomeration of the reinforcement and matrix phases, creates a debonding effect of the fiber and epoxy matrix material as a consequence of the increasing fiber weight percentage. Increasing the fibre content is the main reason for reducing the composite sample's mechanical strength because the fibre content is increased [27]. As a result, it has been found that the pineapple leaf fibre composite sample has the capacity to achieve a higher mechanical strength than previous samples based on sisal and coir fibres, but the results are not as promising (Fig. 3) [28].

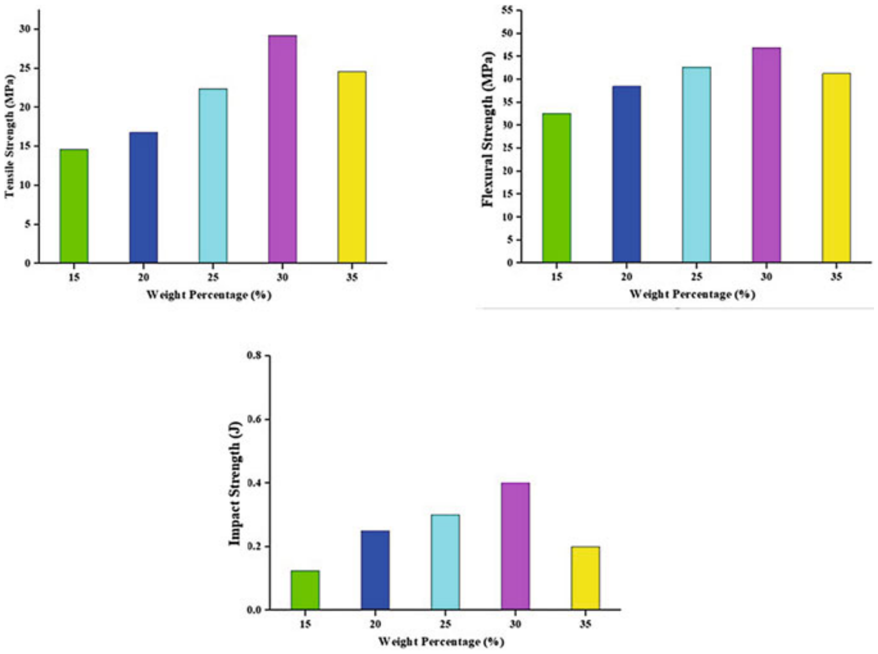

Fig. 3 Mechanical properties of pineapple fibre reinforced composites

3.2 Absorption Studies

The ASTM standard ASTM D5229 is adopted for the studies on water absorption of a composite material composed of water hyacinth fibres and eggshell filler material, while ASTM C413-18 is followed for the studies on chemical absorption of a composite material composed of water hyacinth fibres and eggshell filler material. Depending on the applications, it may be recommended that absorption tests be conducted to determine whether the composites have properties that are hydrophilic and hydrophobic in nature. Using pineapple leaf composite as the main component of a product that will have higher mechanical strength is the main purpose of this research project. A maximum of 50 h are allowed to pass before the absorption test is conducted. As soon as the composite sample is taken from the machine, that sample's weight is checked and reported on every five-hour interval. It was chosen for this absorption study since it has a higher mechanical strength (30wt% of fibre reinforcement) than the composite sample. In the first step, a pineapple reinforced composite sample (30%) of dimensions 20 × 20 × 3 mm is cut in accordance with ASTM D5229 standards to ensure that it meets the requirements. In order to determine the weight of the pineapple composite sample, a digital weighing balance machine is used to calculate the initial weight of the sample. The composite sample (from the water absorption analysis) was immersed in 100 ml of distilled water, and consequently, the composite sample (from the chemical absorption analysis) was immersed in 100 ml of 0.25 molar sodium hydroxide chemical solution.

After the sample has been removed from both the solution and wiped clean with a dry cloth every five hours, it is recommended that the weight of the composite sample be measured after the new weight of the composite sample has been measured after the sample has been removed from the solution and wiped clean with a dry cloth every five hours. In order to test the effectiveness of the monitoring program, there is an initial 50 h monitoring period followed by an additional 50 h monitoring period. As a result of the results of the two absorption studies that were conducted on three samples of fibre reinforcement of different weight compositions, the average value of the absorption coefficients for each fibre reinforcement is presented in this study. From the results of the absorption tests conducted on the composite material, both water and chemical solutions did not appear to have a detrimental effect on it in terms of the effect they had on the composite material. A hemp or flax reinforced pineapple leaf fibre composite, which is made of pineapple leaf fibres, may have a different absorption rate compared to hemp or flax reinforced pineapple leaf fibre composites, which are also made of pineapple leaf fibres. It is due to the fibre content that is present within the pineapple composite test samples that water and chemical solutions are absorbed very slowly at the beginning of the test due to the fibre content within the samples. During the absorption of the solution, the weight of the sample gradually decreases as the solution is absorbed and the sample's weight gradually increases. The fibres reach a saturation point within a certain period of time where they go from absorbing water and chemical solutions to not absorbing them any longer and stop absorbing them after a period of time. At the point at which fibres reach saturation, their life cycle is coming to an end, and this is the time when they reach saturation. There is no change in the weight value of the composite sample once the saturation weight percentage is reached, it remains constant at that point.

3.3 SEM

A scanning electron microscope can be used for examining fractured surfaces and testing samples that are reinforced with pineapple leaf fibre composites under scanning electron microscopes. It is necessary to remove moisture from composite samples for 24 h by using a hot air oven (64 °C) in which they are heated to 64 °C. The epoxy secondary phase material adheres well to the primary reinforcement, as shown in Fig. 4a, indicating that there is good adhesion between these two materials [29]. In Fig. 4b, c, one can see a good bond between the fibres and the matrix. It is possible to use this method as a way of improving composite materials. The pineapple composite sample, as shown in Fig. 4d, shows an unwanted impurity in the reinforcement phase, which is evident on the surface of the composite sample.

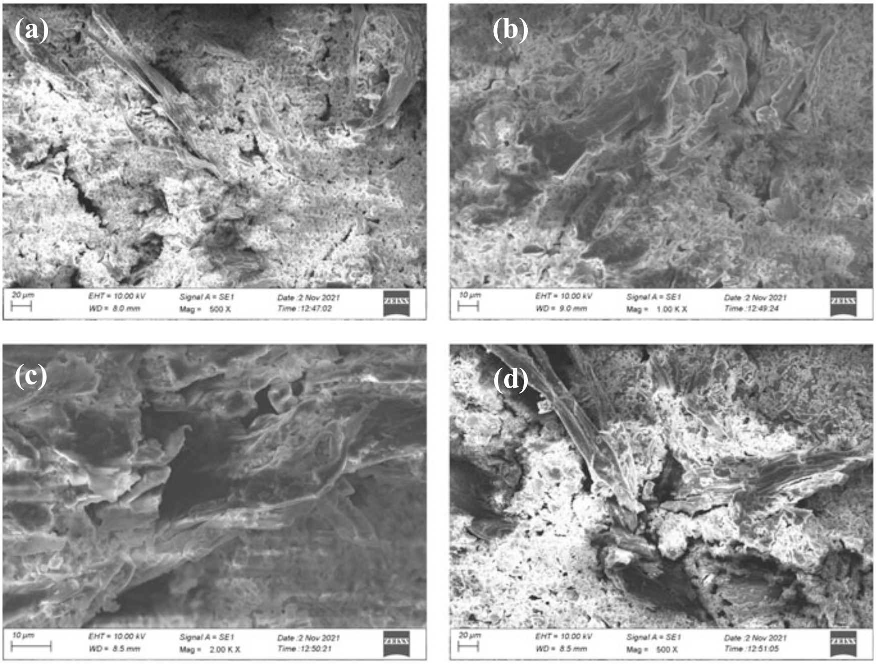

Fig. 4 Surface morphological characteristics of pineapple composite

4 Conclusion

The pineapple leaf fibre with high performance epoxy polymer matrix composite samples properties are identified by this research work.

- The pineapple fibre reinforced composite samples produced by using hand layup method.
- Based on the mechanical strength results the 30% of pineapple leaf fibre reinforced composite samples attained high mechanical strength compared to the other samples (Tensile, flexural and impact).
- After adding the 30% of fibre reinforcement the high weight percentage of pineapple fibre creates the agglomeration effect to the samples. So, the 30% of the pineapple leaf fibre reinforced composite samples have the optimum sample.
- For the absorption studies also, the 30% of the PLF composite samples absorbs very low amount of water compare to the others.
- By the help of scanning electron microscope, the primary and secondary phase bonding and other surface characteristics are easily identified.

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Effect of Interfacial Bonding Characteristics on Impact Strength of Jute Fiber Reinforced Composites

G. Rajamurugan and Prabu Krishnasamy

Abstract Natural fiber-based composite materials have emerged applications in automotive, aerospace, and marine applications. For better mobility, light and strong material requirement led to the development new composites for automobiles and the aerospace industry. This study deals with different composite plates made of different layers of natural fiber (Jute) and resin (epoxy and hardener). The mechanical properties, such as the impact strength of the composites, were studied by introducing different fiber orientations. This chapter reported on gaining data from impact strength and wire mesh reinforcement. The impact strength variations for Charpy and Izod test methods were observed for 30, 40, 50, and 60% fiber volume fractions. The results show that the 60% addition of jute fiber loading improves impact strength by 175%. The impact strength of 45° oriented wire composite specimen depicts better interfacial bonding between the jute fiber and wire mesh than 90° oriented wire mesh composite.

1 Introduction

The superior characteristics of composites increase their significance in engineering. Significantly, the automobile industry focuses on developing lightweight vehicles, recycling, lowering CO_2 emissions, and enhancing fuel efficiency. Composite materials can meet the high strength-to-weight ratio requirements of the automotive and aerospace sectors. Alternatives to classic metallic design that are more accessible and affordable include composite production. According to new environmental standards from the perspective of the material, it is essential given the rising demand for environmentally friendly green material.

Variation in mechanical properties is usually more significant for natural fibers [1]. Natural fiber reinforced polymer composites could be an alternative to more

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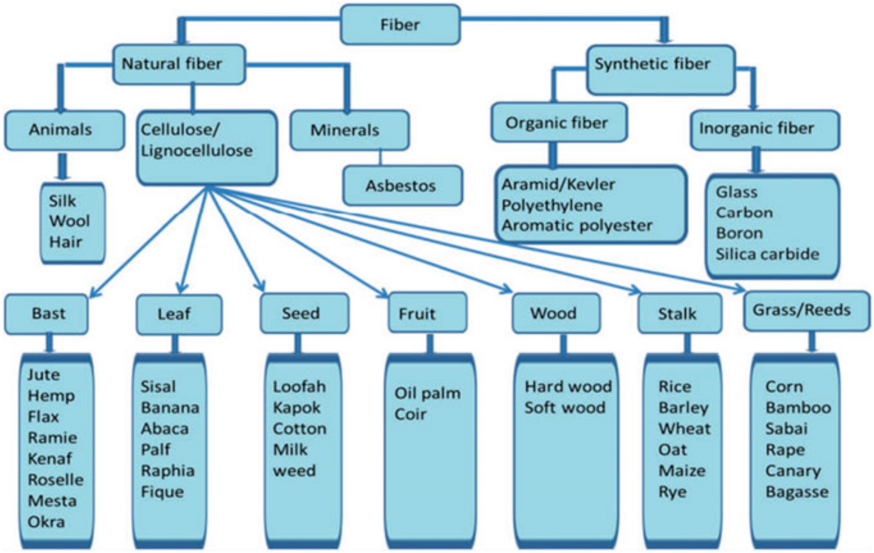

Fig. 1 Classification of fibres [10]

conventional materials like glass and carbon fibers. The properties of carbon fibers are superior to those of natural fibers, but due to their cost and availability, natural fibers are widely used [2]. In general, natural fibers are biodegradable, and these fibers are derived from renewable resources, making them an alternative to carbon fibers (Fig. 1). Natural fibers exhibit excellent mechanical, thermal, low-cost, and availability properties. Natural fiber composites have low energy absorption because natural fibers have a low density. Another high-density material should be used to increase the energy absorption of natural fiber reinforced polymer composites [3]. Among natural fibers, jute fibers have sufficient strength to replace synthetic fibers [4]. The percentages of cellulose, hemicellulose, and lignin in jute fiber are used to determine its physical properties [5]. The weaving direction of the jute fiber primarily influences the mechanical characteristics.

The hydrophilic nature of jute fiber influences the tensile behaviours of natural fiber composite [6]. Compared to mono-polymeric composites, hybrid composites shows better stiffness and strength [7]. Low modulus and inexpensive fibers make hybrids more prone to spoilage. Hybrid composites can provide higher impact resistance, fatigue resistance, and fracture strength [8, 9].

1.1 Polymer Matrix Composite

Especially in polymers, the matrix, which is essentially bonded to the reinforcement, is sometimes referred to as a resin. Polymer matrix composites are classified into two

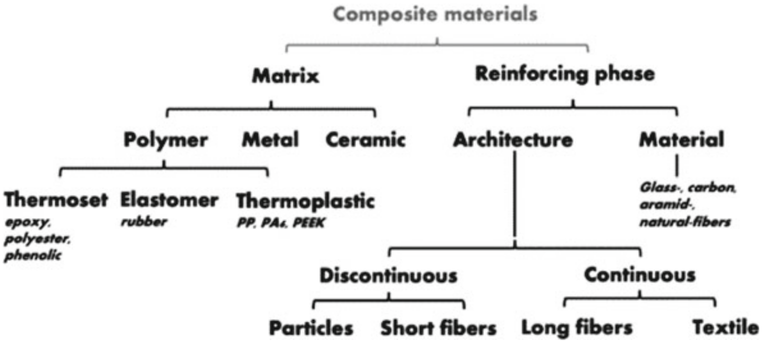

Fig. 2 Classification of composites according to matrix & reinforcements

types: thermoplastic and elastomer composites and these composites are differentiated due to their lightweight (Fig. 2). The cross-linked structure of thermoset resin at the time of curing may result in an un-moldable complicated product. Compared to thermosetting plastics, thermoplastics are remelted and reheated several times. It is also widely recycled and molded into new products. The thermoplastics can resist high temperatures because the temperature may not affect the structural rigidity of the product. Typical examples of thermosetting polymer matrices are vinyl ester, cyanate ester, polyimide, Polyester, epoxy, phenol, and polyurethane.

Similarly, the following resins are typically used as thermoplastic matrixes PEEK, thermoplastic polyimide, thermoplastic polyurethane, polyamide, polyethylene, polypropylene, polycarbonate, PLA [11]. The vulcanization of elastomers may create a cross-linked structure. Rubber is the best example of elastomers; rubber composites are often called elastic composites. The elastic behavior of elastomers is primarily differentiated from thermoset and thermoplastics. Fiber-reinforced tubing, truck tires reinforced with wire or mesh, and aramid fiber-reinforced automobile tires are typical examples of elastomeric composites [12]. In recent days the integration of CNTs into rubber composite may replace conventional fillers like mineral and carbon black [13].

Fiber Reinforced Composite (FRC) can reduce the weight of structural components, mainly in transportation. It is possible because FRC is a premium lightweight material with better mechanical characteristics than steel or aluminum. Most of the FRCs have long fibers reinforced in a polymer matrix. A schematic diagram of FRC is shown in Fig. 3.

1.2 Jute Fiber Polymer Composite

Genus corchorus plants are used to produce jute fiber, and it comes from the family of Malvaceae. Jute fiber primarily consists of lignocellulose fiber, a part of textile and wood. Lignocellulose fiber is one type of libe fiber extracted from libe or bark

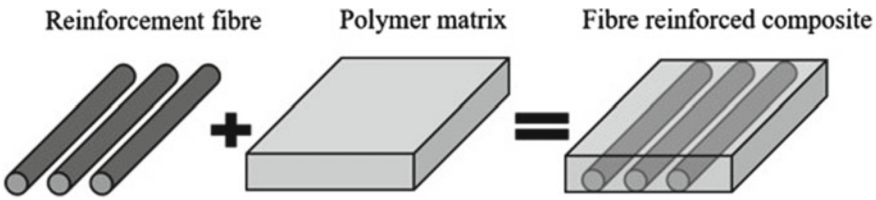

Fig. 3 General arrangement of Fiber-reinforced composite [14]

trees (Fig. 4). The jute fiber contains 11.8% lignin, 0.2% pectin, 64.4% cellulose, 0.5% wax, 12% hemicellulose, and 10% water. Jute fibers hold numerous cells extracted from crystalline microfibrils; the cellulose has a generous layer of amorphous lignin and hemicellulose (Fig. 5a, b). The cellulose and lignin/hemicellulose possess multiple layers. The primary cell wall and the three layers of minor cell walls stick to every layer to form a multilayer composite. The cell layer has different chemical ratios, such as cellulose to lignin/hemicellulose, and different alignment of the cellulose microfibrils [15].

The requirement for natural fiber composites is increasing daily in the automotive and aerospace industries. Natural fibers' mechanical, thermal, and acoustic properties compete with synthetic glass fibers. Natural fibers' biodegradable and recycled behavior are mostly preferred for economy and sustainable development. Natural

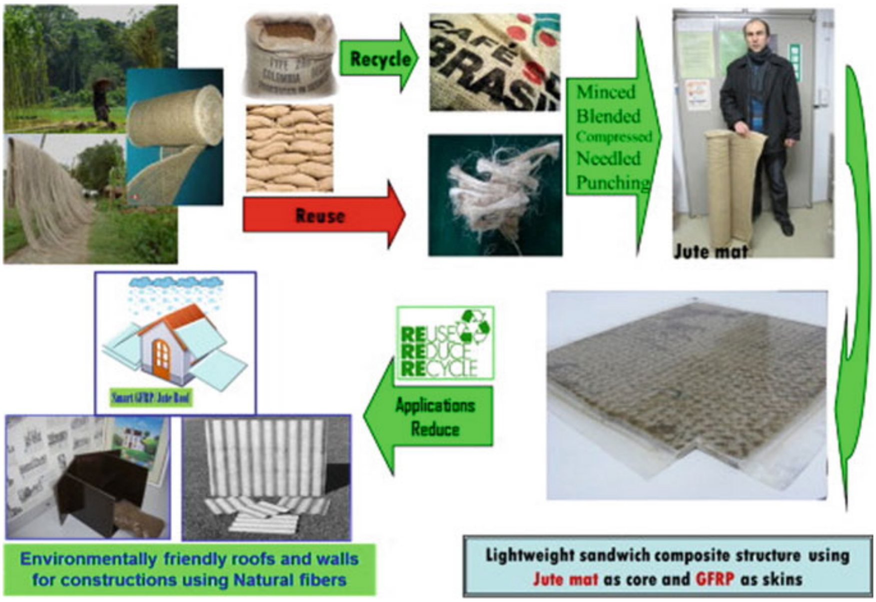

Fig. 4 Overall process of Jute fiber for some potential applications [16]

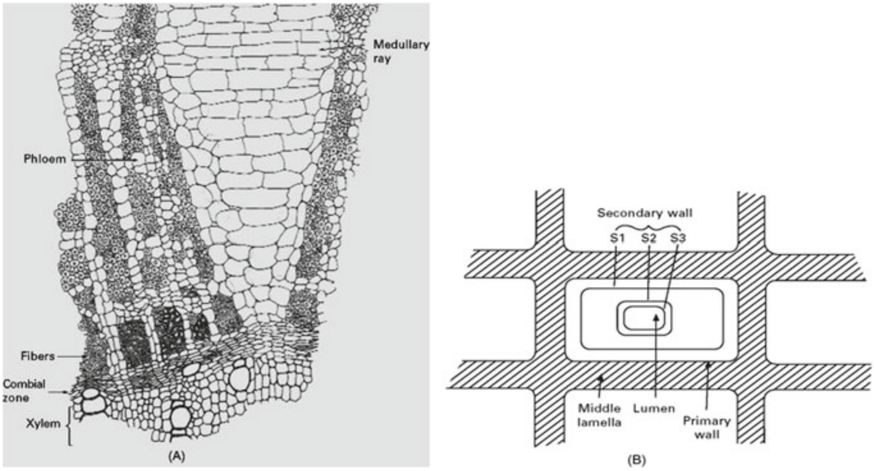

Fig. 5 **a** Cross section of jute stem (Corchorus olitorius); **b** Schematic diagram of jute fiber unit cell [18, 19]

fiber composites are considered green composites when the polymer matrixes are biological in nature.

Nowadays, high-quality surface finish is preferred by most industries on NFC products, and that is not easy to achieve by conventional processes such as thermal compression processes. The heat compression technique produces a low-quality surface finish at the edges. After that, the products are re-machined to get the desired shapes. The thermal compression process is unsuitable for making geometric structures such as mounting holes. So, conventional machining is essential to fabricate the NFC part for the industrial need. Many variations between the microstructure of natural and synthetic fibers can be observed. Hence, natural fiber thermomechanical behavior is not unique to glass fiber composites. So, more analysis is required on the machining part of NFC.

Jute fiber is an environmentally friendly, recyclable, and biodegradable material and it has good acoustic and thermal properties. [14, 16]. 3.2 million tons of jute yarns are produced per annum and used for different purposes. The sack industry is utilizing more amounts of jute yarns. The utilization of eco-friendly Jute bags replaces non-biodegradable polythene bags. In the final stage, the expired jute bags waste is used as landfilled in the form of used sacks or burlap [17]. The physical properties of jute fiber is shown in Table 1.

Table 1 Physical characteristics of natural jute fiber [18]

Cellulose (wt.%)	Spiral angle (degrees)	Cross sectional area, $\times 10^{-2}$ (mm^2)	Cell length, *L* (mm)	*L/D* ratio
61	8.0	0.12	2.3	110

2 Type of Impact Test

An impact test evaluates the toughness property of the material. It is the energy absorbed by the material due to sudden loads. Most natural fiber composites are subjected to impact failure throughout their life, which can decrease their structural stability and load-handling ability. The impact resistance of the composite material is mostly determined by the total number of fibers as well as the type of impact test that is used. The impact strength increases proportionally with the amount of fiber present in the material, but this is applicable only to reach the critical volume fraction. The impact test is classified into four different types [20].

- Low-velocity impact test (velocity—0 to 11 m/s)
- High-velocity impact test (velocity—above 11 m/s)
- Ballistic impact test (velocity—above 500 m/s)
- Hyper velocity impact test (velocity—above 2000 m/s)

The impact load may happen at various speeds and energy levels; for example, ballistics (military), bird attacks, fallen tools, storm (weather), lightning, stream debris, and dragging defects. These incidents may occur from low speed to high speed. The different velocities and variations in impact loads can result in further damage to composite materials, which can occur under various atmospheric situations. Figure 6 shows the reaction of composite material under various impact velocities. The higher impact energy produces significant damage (Fig. 6).

3 Effect of Impact Test on Interfacial Bonding

3.1 Short and Long Fiber Composite

The NaOH chemical treatment on jute fibers improved the fiber composite's toughness which was influenced by the bonding between fiber-matrix. The chemical treatment was carried out by immersing the fibers into different NaOH solutions concentrations like 1, 3, 5, 7, and 9 weight percentages. Generally, the fiber treatment was performed for 24 h at atmospheric temperature. After the treatment, acetic acid is used to wash the fibers and clean them with water. Followed by the fiber was dried under regular sunlight for 24 h. The treated fiber is then used as a reinforcing material to develop fiber-based (jute/epoxy) green composites. The schematic diagram of charpy test specimen was shown in Fig. 7. The impact strength variations for Charpy and Izod test methods were observed for 30, 40, 50, and 60% fiber volume fractions (Fig. 8).

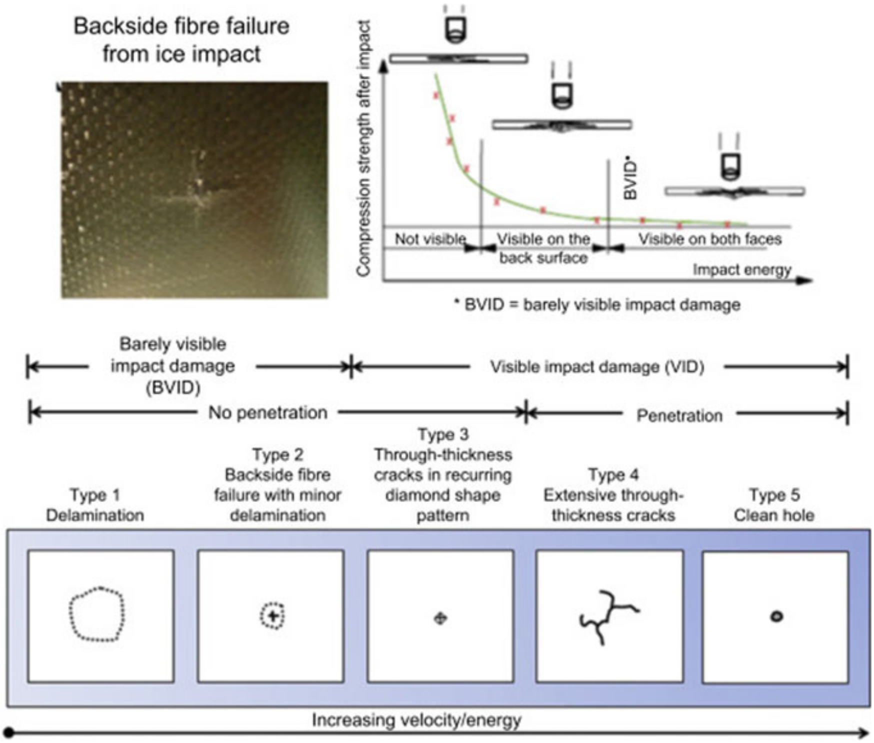

Fig. 6 Failure of composite structures at different impact velocities [20]

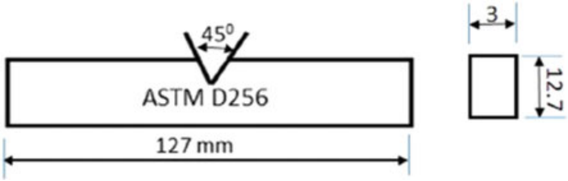

Fig. 7 Charpy impact test specimen

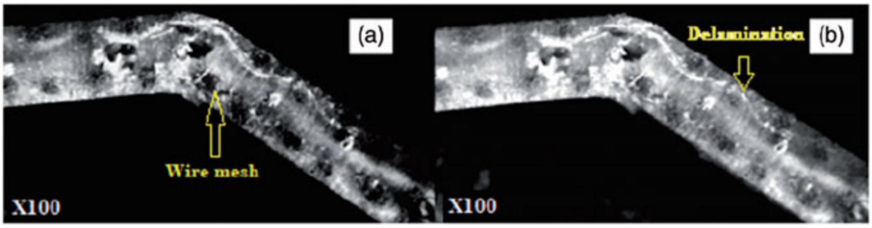

Fig. 8 Interfacial bonding between fiber, wire mesh, and epoxy (**a**). 45° wire mesh (**b**). 90° wire mesh [21]

3.2 Influence of Fiber, Matrix, Fillers, and Other Reinforcement

Impact strength is measured as the capability under resistance to breaking under applied load at high speeds. Izod and Charpy techniques are generally used to estimate the impact strength of jute fiber reinforced composites. The 60% addition of jute fiber loading improves impact strength by 175% [20]. Figures 9 shows a certain improvement in the strength observed in composites with large processing time and the mass fraction of fibers. The processing period increases the brittle characteristics of the treated fiber composite more than the brittle qualities of the untreated fiber composite. Table 2 displays the impact strength of the jute fiber composites.

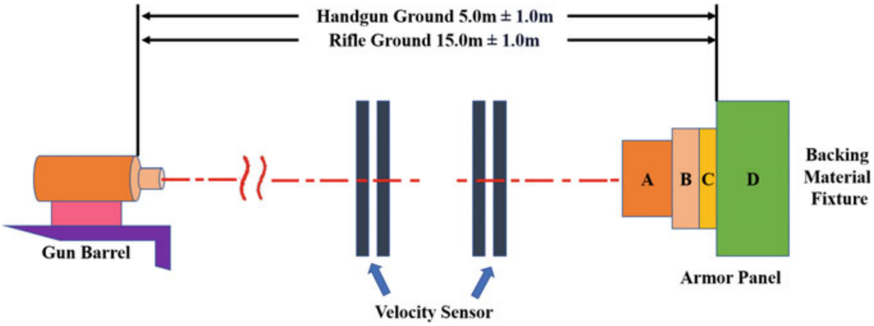

Fig. 9 Experimental setup of ballistic impact

Table 2 Impact strength of jute fiber composites

Author's	Composite type	Effects	References
Govindhasamy K et al.	Jute/ Polyester/ nano clay	15% jute fiber fraction improves the impact strength. Further addition of fiber reduce the strength	[22]
Tapan Kumar Patnaik et al.	Jute/ Epoxy	Short fibers (Randomly oriented) in an epoxy composite have increased the impact strength	[23]
Athith D et al.	Jute/Sisal/ Epoxy/ WC filler	WC filler addition (0–5% & 8%) significantly enhance the density impact strength	[24]
Priyadarshi Tapas Ranjan Swain et al.	Jute/ Epoxy/ Al_2O_3	Fiber & filler addition increases from 0–40% void and agglomeration improved	[25]
Zamurrad Arshad et al.	Jute/epoxy	During drop impact alternative layer of jute & carbon fiber stacking absorbed 1081.7 N	[26]

3.3 Low-Velocity Impact Test (Velocity—0 to 11 m/s)

The jute fiber reinforced composites are growing in attractiveness, their utilization in structural applications is hampered by their poor durability. The hybridization is well-known for its effectiveness in enhancing the strength of fiber reinforced composites. The low velocity impact is an important study to measure the impact resistance of the jute fiber composites under the velocities below 10 m/s. The impact test performed either drop weight test or swing mass test. During testing, the impact force induced damages in the form of dents and visible cracks on the jute composite surface. This is due to matrix failure, fiber delamination and fiber breakage. Several studies are reported the low velocity impact performance of the fiber reinforced composite, however limited study has been found for jute fiber composite. Mahesh et al. [27] investigated about the different stacking sequence of jute rubber composite which has influenced the energy absorption behaviour during conical shaped low velocity impact test. The alternative arrangement of jute and rubber recorded damage resistance for 5 layer and 3 layer provides better energy absorption. The peak force, the coefficient of restitution, and the energy loss percentage were examined, and the failure mode was estimated using microscopic examination. Jute/ rubber composites were revealed to be delamination-free, according to the results of this investigation, owing to matrix breakdown rather than cracking.

Patil et al. [28] analyzed the composites performance from the impact energy absorption of hemp and jute fiber epoxy composite. Using a five-level Central Composite Design with a two-factor model, the impact properties of composites were investigated to determine the optimal input conditions for damage. It was reported that jute /epoxy reinforcement composite absorb more energy than hemp/epoxy reinforced composite. Ultimate loads of 5.90 MPa and 10.68 MPa were determined for hemp and jute fibre laminate, respectively. The low velocity impact performance and damage behavior of hybrid (jute/glass) fiber composite were investigated by Ahmed et al. [29]. The hybrid composites were developed using a glass mould with marginal pressure at ambient temperature by hand layup technique. The test samples were prepared at the size of 150 × 150 mm. Using data gathering software, load-energy-time graphs for four different impact energies were created by varying the drop velocity. After impact testing, the cracks, dents, delamination and other damages present in the tested samples were studied through C-scan. The result found that mono jute fiber composite holds higher enegy absorption capacity than the hybrid jute/glass fiber composite (Table 3). At the same time hybrid composite finds better tolerance capacity. According to the findings, the jute fiber laminates have a higher capacity for absorbing the impact energy of blows than hybrid (jute/glass) laminates; but, their ability to withstand damage is significantly lower than that of hybrid (jute/glass) laminates.

Table 3 Low-velocity impact for various jute fiber hybrid composites

Reinforcement	Inferences	Author details	Year	References
Jute/Glass	Jute fiber composite absorbs more energy than hybrid (jute/glass) composite but has lower damage tolerance	K. Sabeel Ahmed,	2007	[30]
Jute/UP	Higher load resistance at 30 °C is observed	H. N. Dhakal	2014	[31]
E-glass, basalt, jute & flax	E-glass/basalt & E-glass/flax combination revealed higher impact force than E-glass/jute	Muhamad Shahirul Bin Mat Jusoh	2017	[32]
Jute/ Rubber	Regardless of layering sequence, energy absorption is nonlinear	Vishwas Mahesh	2019	[33]
Jute	The impactor radius increases contact force, impact duration and beam indentation. Increased impactor velocity, maximum contact force, and increased beam were discovered	Manar Hamid Jasim	2021	[34]
Jute/Glass	Jute reinforced composites exhibited better energy absorption, and higher damage area whereas, the hybrid (Jute/Glass) composite has high energy absorption, compression strength and minimum damage area	Amuthakkannan Pandian	2022	[35]
Hemp and Jute	Jute fiber composite showed better impact energy absorption than hemp fiber composite	Shreekant Patil	2022	[36]

3.4 Ballistic Impact Test (Velocity—Above 500 m/s)

Ballistic impact is a complex mechanical process that is heavily influenced by the thickness, toughness, strength, ductility, density, and projectile properties of the target material. The utilization of natural fibers and their composite laminates has expanded to encompass a wide range of impact-related applications due to the advancement of durable, strong, and stiffness. The effectiveness of natural fiber composites used in the armour systems was determined by their ability to absorb kinetic energy, their depth of penetration, and the speed at which their impact was absorbed. In especially for ballistic performance in combat, body armour has provided good protection. The most important factor in the design and selection of the material was the enhancement in the body armor's ability to reduce the impact force induced by a projectile's impact by having higher energy absorption properties, while also keeping in mind the importance of reducing its weight. Hard body armor is currently used to guard against impacts that happen quickly. Numerous body armor systems with improved ballistic performance have been developed to shield against such impact. Fiber-reinforced polymer matrix composites are well-known for their strength and stiffness, and they were created to withstand significant energy absorption [37]. The experimental set up of ballistic impact test was shown in Fig. 9.

In addition to the unique qualities of the material (fiber), several mechanisms should be considered in order to improve the ballistic performance of the finished ballistic target. For example, adding extra layers in different configurations during the development of ballistic panels is a popular strategy to improve ballistic protection against ballistic impact in addition to choosing the right material characteristics. [38–42]. According to studies [43] natural-material composites may be employed for ballistic reasons. Jute is a natural fibre that is biodegradable, has exceptional properties, is inexpensive, and is widely available. As a result, they are beginning to replace standard synthetic fibres in composites [44, 45]. Matrix materials such as epoxy and polyester are used frequently, although jute/epoxy composites are more popular because of their superior performance [43]. It has been discovered that a large amount of energy may be absorbed by jute fibers and powdered coconut shell reinforced epoxy composites [46]. Wambua et al. studied the effectiveness of jute-reinforced polypropylene sandwiched between sheets of mild steel. When compared to plain steel or jute composites, the performance of the sandwiched composites was superior [34]. In a study conducted by Fernanda Santos da Luz et al., an investigation was carried out on jute fabric composites and the results revealed that jute fabric composites exhibited superior ballistic performance compared to aramid fabric composites [45].

The ballistic impact study focuses on the material failure behaviour caused by projectile/bullet impact. It is especially important in nuclear and military applications to protect both people and equipment. The performance of the composites under impact conditions is influenced by various factors, including the size, shape, mass, impact velocity, and material properties of the projectile [46, 47]. Additionally, the geometry, mass, composition, and support condition of the target also play a significant role in determining the impact performance of composites [48]. Ballistic impact testing is performed on the Jute epoxy composite plate and Jute-rubber epoxy composite plate. The two chronographs are used to record the input and residual velocities after bullets are fired from a distance of 5 m at fixed samples. Thicker jute epoxy plates are more effective at absorbing energy because their residual velocities are lower. The impact and residual velocities exhibit a linear relationship, as shown in Fig. 8a. As illustrated in Fig. 8b, thicker plates absorb more energy. When increasing plate thickness from 3 to 15 mm, there is a nearly 80% increase in the amount of energy absorbed. Jute epoxy plates could be made thicker to make them more bulletproof. The sandwich plates absorbs far more energy and has lower residual velocities [49]. When a bullet completely breaks through a jute epoxy plate, a hole will be clearly apparent. Jute epoxy plates broke easily and frequently due to their brittleness.

4 Applications

Commercial applications of the current state of the art necessitate composite materials with better thermal characteristics. Around the world, people are using natural fiber-based goods in textile applications, wooden brushes, woven fiber mats, and other fiber products. Recently, the composite reinforced by natural fibers has found many applications in modern vehicles. Specifically, it can be found in body panels, handrails, center liners, top door support panels, hand rest, and passenger seat cover panels. The fiber/polypropylene composite material offers better functionality and durability in the vehicle's rear deck.

The rear panels and package trays are composed of natural fiber composite materials to reduce engine and road noise inside the vehicle. Components such as door cladding, seatback lining, and floor panels are developed from hemp, sisal, and flax fiber composites. The several application of bast fiber reinforced composite are listed in Table 4. Since natural fiber composites are recyclable and do not affect the environment, many studies have tried to develop car parts and components composed of these materials. Other significant industry areas are currently moving toward composite parts, taking the environment and recyclability into consideration. Recently, manufacturing industries have adopted new technologies to recycle heterogeneous trashes and scrapings to favor the exponential development of natural fiber composites.

Table 4 Automotive applications of Jute fiber polymer composite

Author's	Composite type	Fiber type	Inferences	Automotive application	References
James Holbery et al.	Hybrid composite	Jute, Sisal, and hemp	To commercial products several technical constraints to be addressed	Door covers, seat back panels, package covers	[50]
Suriya Ferdous et al.	Hybrid fiber composite	Jute/kenaf	New automotive application of jute fiber composites improves the market share of jute fiber around the world	Front door liner, Boot liners, Parcel shelves, Soundproof shields, Headrest	[48]
Layth Mohammed et al.	Fiber composite	Jute and flax	The requirement of natural fiber composite in automobile industry is comprehensive discussed	Truck liners, door-trim panels & car interior parts	[51]
Krishnasamy P et al.	Hybrid composite	Jute and hemp	The fiber composite reduce the CO_2 emissions	Headliner and body panels	[52]

5 Summary

The impact strength of jute fiber is improved by the inclusion of filler, and this improvement is further attributable to the unsystematically short fiber composites. The addition of wiremesh reinforcement significantly increased the impact strength up to 15–20%. The hybrid fiber reinforced polymer composite improves the general performance compared to mono fiber reinforced polymer composites. The performance of the composite can be significantly improved by selecting appropriate hybridization procedures, including the use of hybrid materials, various fiber layers, specific stacking sequences, and the reinforcement of fillers.

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Effect of Interfacial Bonding Characteristics on Dynamic Mechanical Analysis of Cotton Fiber Reinforced Composites

Dalila Hammiche, Rebiha Bellache, and Amar Boukerrou

Abstract The use of composites continue to grow thanks to low cost processing, ecofriendly and design flexibility mainly when natural fillers are used as reinforcement. These later are widely used as reinforcement for polymers due to environmental concerns as well as for providing high performance. An interphase is created between the two constituents and will have the role of transmitting the constraints from one to the other, hence the establishment of good adhesion. The interfacial adhesion is very important to improve the properties of composites. In this chapter, the use of cotton fibers as composites in the strengthening phase will be reported. Next, the different treatment methods used to improve the interfacial bonding between the fiber and the matrix and its impact on the dynamic mechanical properties of cotton fiber reinforced composites will be debated.

Keywords Composites · Cotton fiber · Interfacial adhesion · Dynamic mechanical analysis

1 Introduction

Composite materials are obtained by the physical or chemical combination of two or more different constituents. The application of composites reinforced with natural fiber is increasing. These fibers are added to improve many properties of these materials. More than, natural fibers are renewable materials with lower greenhouse gas emissions thus environment respectful [25].

Tusnim et al. [52] prepared composite based on polypropylene reinforced with jute and sheep wool fibers. It was observed an improvement in mechanical properties and thermal stability. According to the authors, the use of natural fibers procures environment friendly materials and the required properties.

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Bolcu et al. [12] studied the mechanical characteristics of composite materials reinforced with flax fibers. The results show that the composite materials under investigation may be used in aerospace, building and civil and industrial engineering.

Indeed, natural fibers as reinforcement for polymers are used due to environmental concerns as well as for providing high performance, and processing advantages at favorable cost [23].

Natural fibers are generally arranged in three categories: plant, animal, and mineral fibers. Vegetable fibers gain more attention than animal fibers. Animal fibers have different type of functional amino acids whereas vegetable fibers composition is mainly cellulose, hemicelluloses, and lignin [34].

Cellulose is the largest biological molecule on the planet. From a chemical point of view, cellulose is a macromolecule made up of a very long stereo-regular chain of $C_6H_{12}O_6$ glucose links. The unity of repetition is the cellobiose dimer. The amount of repetitive units or the degree of polymerization varies according to the origin of the cellulose. This polymer is of great interest from an industrial point of view since many fibers (cotton, hemp, jute, linen…) are composed of cellulose. Cellulose has a fibrillar and partially crystalline structure, the microfibrils of cellulose are composed of ordered crystalline areas and amorphous areas totally disordered, Mitra et al. [42] therefore, Cellulose is primarily the fibre reinforcement material and the quality of the reinforcement may still depend on the micro fibril angle and crystallization.

Hemicellulose is the second most common polysaccharide and contains 17% to 35% dry matter lignin [30].

Hemicellulose has emerged as a viable alternative for agricultural waste due to its usefulness in numerous agro-industrial processes. For instance, hemicellulosic biomass can be converted into fuel or chemicals [46].

Lignin is a polyphenolic amorphous substance mainly derived from the dehydration radical polymerization of monolignols [20].

The properties of the extracted lignin (technical lignin) depend on both the biomass resource and the extraction process [9].

Many plant fibers are used as reinforcing materials. Through this chapter, relevant background of the impact of interfacial bonding on dynamic mechanical behavior of cotton fiber (see Fig. 1) reinforced composites will be addressed.

There have been several studies describing the use of cotton in polymer as reinforcement. Sutivisedsak et al. [48] investigated the properties of PLA reinforced with cotton. It was observed a broad exothermic cold crystallization peak, and a low degree of crystallinity suggesting that cotton fibers are acting, as nucleating sites and there here are some interactions between fibers and matrix that enhance cold crystallization.

Cotton is the foremost well-known fiber utilized to create a wide run of textiles. Cotton textiles are used widely because they are breathable, lightweight and flexible. Cotton's hydrophilic nature is advantageous in a variety of applications. However, due to potential uses in outdoor clothing and protective textiles, the engineering of super hydrophobic textile surfaces is gaining significant scientific and industrial interest [39].

Fig. 1 Cotton plants in Punjab, India

The utilization of cotton dates well from 7000 years; Cotton is a fiber that grows in a boll or protective case around seeds on cotton plants (Fig. 2). It is soft, fluffy, and stapled. Around 26 million tons of cotton were produced worldwide for the season 2019/2020 and India, China, United States are the leading cotton producers.

The main cotton compounds are cellulose (95%), the remaining is hemicellulose, pectin, wax, and water (see Table 1) and Cellulose type and degree of crystallinity influence reinforcement effectiveness [11].

Fig. 2 Cotton fiber harvest, Punjab, India

Table 1 Chemical composition of cotton

Cellulose	Hemicellulose	Pectin	Water soluble	Wax	Water
82.7%	5.7%	5.7%,	1.0%	0.6%	10%

Cotton fiber has a degree of polymerization of around 7000, low density with higher strength and impact resistance and absorption [15].

Important mechanical characteristics of cotton fibers can be found in Table 2.

The mechanical properties of the neat polymer are lower than composites manufactured with cotton fibers. Borsoiet al. [13] investigated the use of cotton fiber as reinforcement in composite materials. The results suggest that mechanical behavior was satisfactory. There was an increase in flexural modulus and flexural strength, and a high impact. The study of [28] suggested that with the mixture of cotton fiber with polyester, there was an enhancement in the capacity for structural stability of the composite, and a higher coefficient of friction as well as the reduction of the contact-surface-temperature for this composite.

Tasdemir et al. [50] produced composite by using polypropylene with cotton waste as reinforcement. According to the obtained results, hardening values and flow resistance was found stable whereas Izod impact values were raised. However, SEM result show the presence of halls between fibers and matrix surface. This is due to the absence of compatibility hence; there was low adhesion between the fiber and the polymer.

Composite materials include at least a matrix and a fiber, which are separated by an interface phase. A difference in polarity between fiber and matrix causes incompatibility thus an ineffective stress transfer that yields weak properties. The chemical treatment may ameliorate the adhesion of the matrix to fiber; in addition, the stress transfer can improve overall properties [25].

The difference of chemical structures between the cotton fiber and the matrix requires a treatment when the composite is manufactured in order to improve the compatibility between them. For instance, SEM analysis (Fig. 3) indicated the formation of cavities on the surface of PVC/cotton composites resulting from pull out of fiber owing to wetting insufficient of cotton to polymer. Indeed, adhesion is mediocre. The incompatibility does not promote the bonding and cohesion of cotton fiber to the matrix, thereby advancing the pull-out of fiber and cavities formation.

The chemical treatments are used to reduce the hydrophilic nature of the cotton fiber. This chapter is dedicated to report the impact of interfacial bonding on the properties, mainly thermomechanical properties of composites reinforced with cotton. More detail concerning this point is detailed below.

Table 2 Mechanical properties of cotton fiber [27]

Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
287-597	5.5-12.6	9.7

Fig. 3 Micrograph of cotton fiber reinforced PVC composite surface

2 Interfacial Bonding

Interfacial adhesion mechanisms are among the main predominant aspects that influence the behavior of the composite materials. The parameters affecting the adhesion between the matrix and filler are the molecular attractive forces, chemical bonds, and the electrostatic adhesion [6]. Therefore, this part of our chapter clarifies the importance of interfacial adhesion on the composites characteristic. According to the literature [21], Numerous approaches were used to enhance the compatibility of fiber and polymer and play a fundamental role in the mechanical properties of the manufactured composites. Mainly two ways are used, (1) fiber modification by physical or chemical treatment and (2) the addition of compatibilizer for interfacial adhesion improvement.

The composites development with the best properties requires modifying the fiber surface by using reagents that are capable of bonding to the hydroxyl groups of the fiber. The used compatibilizer forms chemical bonds such as maleic anhydride by interaction with the hydroxyl groups of fiber and in fact of its compatibility with the matrix [51].

Indeed, the compatibility can be improved when chemical modification of the fiber is performed. For this, the humidity degree plays an important role on interfacial characteristics [49].

The most chemical treatment used is esterification because it increases the fiber–matrix interaction and higher strength properties can be reached by using high-ester content [29]. Usually, chemical modifications, like acetylation, and chemical grafting are used to improve the surface properties of the fibers [17]. Sodium hydroxide, acetic acid, silane solution, benzoyl peroxide, polymer coatings, bleaching, corrosion and stearic acid are also used for cotton fiber as chemical treatment [19].

Aruchamy et al. [3] investigated the impact of cotton/bamboo hybrid used as reinforcement. Chemical treatment with NaOH solution was conducted and it was added to epoxy matrix to manufacture of composites by the process of compression molding. After chemical treatment, the fiber surface is rough and all impurities are removed. This enhance the interfacial bonding between cotton and epoxy resin thereby mechanical properties are improved.

Ibragimov et al. [33] studied the chemical modification of plant filler based on non-recyclable cotton and linen spinning wastes with 1% sulfuric acid solution and which increased indicators of heat-insulating of composites of composite heat insulating plates. Moreover, the surface modification increases the strength of the material, when the cotton waste was increased.

Wei Cao et al. [55] studied hybrid composite PET/cotton fibers reinforced HDPE, treated with the synthesized modifier to enhance the composite properties, from the obtained results, the compatibility between treated fibers HDPE was notably ameliorated, resulting in improved the strength.

Alkaline treatment increases the surface roughness and breaks down the fiber bundles that leads to the discount in diameter of the fibers to the micrometer scale and formation of fibrillation, which can create a good adhesion. Alkaline treatment also remove a part of lignin and wax from fiber surface, thus allowing a better anchoring of the fiber with the matrix [10, 43].

Leonard et al. [37] investigated the properties of composites based on PP and kapok/cotton hybrid fiber. Acetic anhydride was used as chemical treatment in order to improve adhesion between matrix and fibers. The results show that acetylated composite materials have a higher onset temperature of degradation thus improved their thermal behavior. The composites reinforced with untreated fibers have less strain at break compared with treated ones.

Calabia et al. [14] used cotton fiber treated with silane as a reinforcement in poly (butylene succinate). It was established that after silane treatment, there is an improvement in tensile strength and a slight enhancement of the thermal stability as well. Furthermore, the use of cotton fiber as reinforcement increase the biodegradation rate of composite materials in controlled compost at 58 °C.

Battegazzore et al. [7] assessed the properties of PLA and PHB combined with cotton fabric using the stacking method. With the addition of an epoxy-coupling agent, the interaction between fiber and matrix can be reached to enhance composite properties. It was observed that cotton improved significantly mechanical properties. The adding of epoxy-coupling agent to the PHB-based composite showed a modulus increase of 15% with respect to the untreated composites.

Zonatti et al. [57] studied the effect of recycled cotton on thermoset resins properties. According to the obtained results, the incorporation of 30% (v/v) of fiber increases approximately twofold compared to neat resin tenacity and Young’s modulus for composites prepared with epoxy resin. Moreover, doubled in the toughness of pure resin and tripled in Young’s modulus for polyester resin composites.

Uddin et al. [53] investigated polyester resin reinforced with jute, cotton and glass fiber. The obtained results showed that the introduction of natural fillers and glass increased the tensile and flexural strength of the material and thereby the improvement of the mechanical characteristics of the resin/cotton, jute, glass composites.

Mengyun [40] fabricated conductive wires with cotton wicks and nanofillers. And from the results, CNT-cotton composite yarns (CNT-CY) showed better tenacity, but decreased the elongation at break.

Abrar et al. [1] invistigated polypropylene-cotton hybrid composite with various percentages, and according to the results found, the average values of coefficient of friction were 0.64, 0.75, 0.88 and 0.94 for neat propylene, 10, 30 and 40% reinforced cotton, respectively. The Rockwell hardness value of the composites enhanced due to the increase in fiber fraction. Based on the coefficient of friction values and hardness, the polypropylene-cotton enhanced hybrid composite could be employed for thermal and sound processing applications in a functional manner.

Ênio et al. [18] studied the mechanical characterization of a polyurethane PU biocomposite based on plant enhanced with alumina trihydrate and supported by cotton. The tensile test revealed an enhancement in mechanical properties. Wei et al. [56] studied polyester/cotton composite. The improvement in the strength is due to the increase in cotton content and to the strong interaction between the matrix and the plant filler.

In another study for the same composite cotton/polypropylene, Abrar et al. [1] confirmed that the thermal and acoustic characteristics depend mainly on the nature of the fibers, the porosity, the voids and the microstructure of the composite material. According to Alomayri et Low [2], a weak bonding leads to the reduction of the impact resistance and the reduction of the mechanical properties. Cotton fiber has a higher tensile strength than polyester resin, which is a reinforcing effect for the composite; these results found by Paiva et al. [44] could be a consequence of the low fiber-matrix interface that typically develops when unprocessed lignocellulosic fibers are introduced into composites.

The study of Paiva et al. [44] revealed that the mechanical properties of an all-cellulose composite enhanced with cotton fabric are based on the correct selection of treatment parameters, i.e., pressure, time, and solution concentration; affect the mechanical characteristics of the composites by their effect on the crystallinity, tensile and elongation strength of the composites. Mechanical characteristics are commonly important for fiber-based composites including static bending, tensile strength parallel and perpendicular to the surface, edgewise shear, shear strength, and glue line shear. ASTM D 1037.

The tensile strength of the composite material manufactured by Balaji and Senthil Vadivu [5] increased linearly with the addition of cotton fiber. The interfacial properties, internal cracks, and internal structure of fractured surfaces of the tested composite samples are noted. Therefore, concluded that the composite material produced with cotton fiber and unsaturated polyester has higher mechanical strength compared to other combinations of coir-cotton fiber enhanced with unsaturated polyester and can be used in wrapping applications.

Interfacial bonding is an intimate contact between fiber and matrix (Fig. 4). The mechanism occurs by either wettability, or chemical adhesion, or mechanical keying [36].

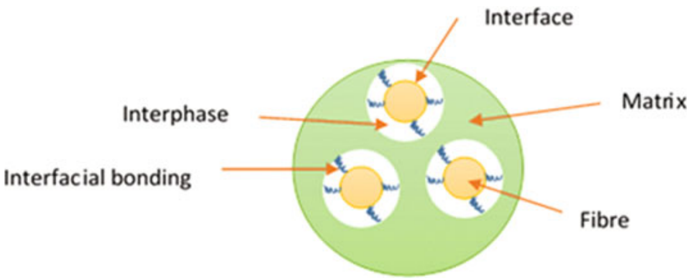

Fig. 4 Schematic of an ideal composite

The measurement of the adhesion degree can be provided by many techniques such as bending, pulling, peeling, laser spallation, and sliding. These technics give key information on the macroscopic state of deformation at the interface, such as the displacement of applied force [35]. The flaw in those methods is that during the fiber pull-out process, the residual stress in the material relaxes and promotes the driving force for further decohesion [16].

The interface fiber-matrix influences mechanical and thermal behavior of composites materials as well as thermomechanical properties.

3 Effect of Interface Adhesion on Thermomechanical Properties

It is mandatory to test and ensure that performances of the manufactured composites achieve the required industrial standards, especially when submissive to periodic stress such as damping [38].

Dynamic mechanical analysis is considered the most used method in the material engineering field to analyze composites and evaluate the viscoelastic behavior in terms of time, temperature, or frequency. Storage and loss modulus and damping factor are the main considered parameters [4].

Dynamic mechanical study is an accurate approach to examining the relaxation behavior of composite. The thermomechanical properties measurement can provide information relevant to the polymer-fiber interface. For instance, the bonding among polymer-fiber is strong when the tan δ peak position shifts. The reduction of damping amplitude points out that the number of molecular segments implicated lowered because of the improvement in fiber-polymer adhesion [22].

It is also stated that the dynamic mechanical analysis notes the dissipation of the energy during relaxing and compression states, throughout a range of temperatures. Higher energy dissipation reflects weak interface adhesion. Damping values are reduced when there is a reduction in polymer chains' mobility due to strong interfacial bonding [36].

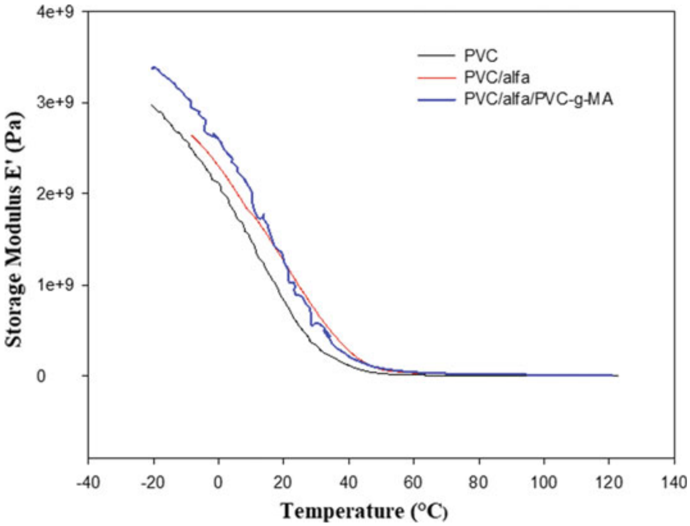

Fig. 5 Storage modulus of PVC and PVC/alfa composites with and without compatibiliser

The dynamic mechanical study outcomes conducted by He et al. [31] indicated that showed that fiber inclusion has a great impact on DMA parameters (storage and loss modulus and damping ratio) due the good interfacial adhesion thus better stress transfer and more efficient energy dissipation at the fiber-matrix interface. Indeed, the work's results conducted by Hammiche et al. [24] show that the storage modulus of the composite is remarkably higher than that of the matrix alone, this may be due to a structure of a network of interconnections of the fiber with the matrix. The effect of adding the compatibiliser into the composites is obvious; it is probably attributed to the existence of a strong interaction at the fiber-matrix interface (see Fig. 5).

Based on an approach presented by Way et al., the addition of cotton as reinforcement in the PLA matrix improved the thermal stability of composite materials. Excellent improvements in both stiffness and toughness were achieved. DMA results denoted that the mechanical loss factor was decreased; suggesting better damping capabilities and good adhesion were achieved [54].

Henrique et al. [32] assessed the dynamic mechanical properties of manufactured composite based on waste cotton and unsaturated polyester. The results show that the values of the peak height of the tan delta were lower, due to better fiber-matrix interaction.

Numerous studies reported that better dynamic mechanical properties of composites could be achieved when fiber hybrid is involved. Hybrid composites are manufactured using more than one fibers in the same polymer matrix to improve the composite properties and establish a synergistic impact [26].

Mirella et al. [41] reported that the mechanical properties of composites based on polypropylene and braided cotton were improved, in terms of elastic modulus and tensile strength; using a thermoforming process. The presence of fiber decreases the density by around of 15%, in addition to the no change in the Tg in DMA measurements.

Kamble et al. (2021) investigated the dynamic mechanical properties of composite based on graphene oxide/cotton fiber hybrid and epoxy matrix. It was established that the incorporation of graphene oxide in the hybrid epoxy composite at low loadings improved the stiffness. According to authors, there is an increase of the damping factor due to poor particle–matrix interface bonding at high rate content of graphene oxide. This demonstrates the properties can be improved when cotton is used at high loading instead of graphene oxide.

Battegazzore et al. [8] studied multilayer composites composed of PLA or PHBV stacked on twill fabric based on cotton. The obtained results for the dynamic thermo-mechanical analysis revealed that there is an increase in heat deflection temperature for the PHB matrix from 70 to 123 °C. In the case of amorphous or predominantly amorphous polymers, the increase in heat deflection temperature is mainly connected to the crystallinity increase, such as PLA.

For this, material annealing leads to an increase in crystallinity, and when it is added to the fiber, it may drastically increase its thermomechanical properties.

According to another study, the silica-cotton aerogel composite prepared using a glass precursor soluble at room pressure by a drying method, the structural strength was improved, therefore, the particles attached to the cotton can controlled the heat transfer [47].

Pinho et al. [45] improved the synthesis of composite dressing obtained by the association of cotton textile substrates and a cyclodextrin-hydroxypropylmethyl cellulose, and according to the obtained results, they have found that with the increase of the temperature reduces the stiffness of the composites. The same remarks were observed in the work of Alomayri et al. [2], where they studied composites prepared with woven cotton fabric, and they found that the compression strength, flexural strength, and fracture toughness of the composite decreased with the increase in temperature, or when the temperature of 600 °C is reached, the composites presented an important decrease in mechanical characteristics.

4 Conclusions

The use of composites continue to grow thanks to low cost processing, ecofriendly and design flexibility mainly when plant fibers are added as reinforcement. In fact, plant fibers are widely used to reinforce polymers due to environmental concerns as well as for providing high performance. As several natural fibers, cotton is added to reinforce a number of composite materials. However, strong bond is imperious at the interface fiber-matrix to achieve better properties. Fiber-matrix bonding can be improved when chemical modification is applied. Indeed, interfaces affects the behavior of manufactured materials. Strong interface induces high properties. A chemical modification leads to a good cohesion and wettability between fiber and matrix thus a better interfacial bonding can be reached. The thermomechanical properties measurement can provide information concerning the properties of composites at the interface fiber-matrix. For instance, the tan delta peak position shifts is an indication of the presence of a strong interactions at the interface fiber-matrix. The fiber adding affects DMA parameters (storage modulus, loss modulus, and damping ratio) due the good interfacial adhesion thus better stress transfer and more efficient energy dissipation at the fiber-matrix interface.

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Effect of Interfacial Bonding Characteristics on Fatigue Behavior of Hemp Fibre Reinforced Polymer Composites

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Abstract Demand for eco-friendly, sustainable and biodegradable natural fiber-reinforced polymer composites (NFRPs) are continuously expanding as global environmental concerns and awareness of renewable green resources continue to grow. Due to their superior physicochemical and mechanical properties, natural fibers already occupied a significant place in the composites industry. NFRPs are widely used in the automobile, aerospace, personal protective clothing, sports, and medical industries as alternatives to costly and nonrenewable petroleum-based synthetic fiber-reinforced composite materials. *Cannabis sativa L.* (Hemp) has received a lot of attention because of its multipurpose usability, short production cycle, low capital demand in cultivation, possibility of carbon-negative transformation, and easy carbon sequestering material. Hemp fiber cultivation and extraction techniques, their physicochemical properties, and technical feasibility for composite structures were discussed

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in this chapter. In addition, diverse types of polymer matrices including synthetic polymers and biopolymers were briefly discussed. The interfacial bonding between fiber and matrix, which determines the ultimate properties of composites, has not been found satisfactory in recent studies. Therefore, a significant amount of research is currently underway to improve interfacial adhesion between natural fibers and polymer matrices. The recent techniques to improve the interfacial bonding and the effects of interfacial bonding on fatigue behavior such as stress–strain hysteresis, strain energy, viscoelasticity, and hysteretic energy dissipation properties of hemp fiber reinforced polymer composites (HFRPs) were precisely discussed. This chapter concluded by mentioning the diverse types of existing challenges associated with HFRPs and providing the necessary future research directions as well.

Keywords Textile structural composites · Fiber reinforced composites · Interfacial adhesion · Polymer composites · Hemp fiber composites · Fatigue behavior

Abbreviations

AAPP	Atmospheric Air Pressure Plasma
FRPCs	Fiber reinforced polymer composites
HFRP	Hemp Fiber Reinforced Polymer composite
IFSS	Interfacial Shear Strength
MA	Maleic anhydride
MAPP	Maleated Polypropylene
NFRP	Natural Fiber Reinforced Polymer
PCL	Poly Caprolactone
PMPPIC	Polymethylene-Polyphenyl Isocyanates
PP	Polypropylene
Tg	Glass Transition Temperature

1 Introduction

Impactful maintenance of environmental sustainability is gaining significant importance currently, which consequently increases the interest in using natural biodegradable materials in the composite industry [114]. Natural fiber-reinforced composites are gaining popularity in diversified sectors like construction, automobiles, firefighting, etc., as a substitute for synthetic and non-degradable composites [95]. Natural fibers are one of the most suitable and environmentally friendly alternatives to traditional synthetic reinforced composites at a minimal cost. The easy availability and long-term durability of NFRC (natural fiber-reinforced polymer) placed it in a growing position in the present market of global reinforced polymer composite material [96]. Easy availability and lower cost are other potential factors that create economic interest in the production of NFRC nowadays [29]. Bast fibers like hemp, flax, jute, sisal, etc., are considered as the potential source of natural fiber while reinforcing it in a composite material for their properties of substituting conventionally used fibers like glass, steel, etc. in the area of polymer composite preparation [42].

Hemp is one of the most suitable fibers for reinforcement in polymer composites. This ecologically favorable fiber can rule the sector of NFRC with its advantageous mechanical properties. The hemp plant has grown predominantly in the Asian region once a year for around 12,000 years. However, the industrial production of hemp fiber occurs minimally due to improper utilization of the benefits of hemp for industrial purposes. Being cellulose-based fiber, hemp also contains hemicellulose, pectin, and lignin in its chemical composition. The chemical composition, mechanical properties, and fiber diameter of hemp deviate according to age, the procedure of retting, country of origin, environment and weather of the origin country, techniques of manufacturing and separation of fibers etc. However, the presence of a notable amount of cellulose made it usable as a potential reinforcing fiber for polymer composites [98].

The higher tensile strength and stiffness of hemp fiber are considered one of the substantial advantages of using it in a polymer composite. In addition, the aspect ratio of hemp fiber is higher, along with lower fiber density. These properties also increase the usability of hemp in NFRP composites. However, the age and gauge length of the fiber impacts its strength. According to the finding of Pickering et al., the strength of fiber elevates deliberately in the flowering stage of the plant, and the fiber with a descending gauge length has incremented strength [78].

The application of hemp fiber as a resin matrix composite was initiated in the automobile industry, a substantial sector of NFRP [98]. Consequently, this fiber started to be used in many other sectors as a form of HFRP. Hemp fiber-based biopolymers can effectively substitute oil-based synthetic polymers. Though these natural biopolymers are entirely biodegradable, they can meet the necessity of maintaining environmental sustainability [55]. Diversified fabrication techniques like lamination [112], micro-braiding [49], etc., are followed to implement HFRP composites effectively.

Hemp fiber also has some constraints as a reinforcing material. One of them is lower adhesion towards the interfacial bond creation with polymer matrix. Interfacial bonding is the mechanism of creating adhesion between the polymer matrix and the reinforced fiber. Strong interfacial bonding results in a better stress migration from the polymer matrix to the reinforced fibers. Consequently, the composite particles become smaller in size which assures better interaction with the surface area of the fabric in a more effective manner [76]. Sufficient wetting is obligatory to achieve an effective interface between the polymer matrix and reinforced fiber. Adequate wetting properties can ensure the effective flow of liquid polymer matrix over the surface of the reinforcing fiber which as a result provides a smoothly bonded polymer composite. Insufficient interfacial bonding between fiber and matrix increases the cracking tendency of the composite material and proportionally reduces the shear strength of the material [99]. Usually, cellulose base fibers are notably hydrophilic, which cannot create adequate adhesion with the thermoplastic molecules as they are predominantly hydrophobic [94].

The hydrophilic nature and irregular surface morphology of hemp fiber increment the tendency of cracking in resin-rich portions of the polymer composite. A weak interface makes the composite brittle which impacts the fatigue behavior directly. Surface modification along with the usage of a hybrid combination of other fibers with hemp can resolve the issues at an acceptable level. Diversified modification techniques can be applied in both matrix and fiber can be an effective solution [99].

Physical modification techniques like ionization of the fluid matrix, treating the fiber surface with plasma and alkali, etc. can improvise the performance of the composite material [46]. According to Ragoubi et al., the tensile strength of hemp and polyester matrix incremented notably while Young's modulus also increased by 30% after treating it with ionization of the fluid matrix [85]. The effect of treating the fiber with atmospheric air pressure plasma (AAPP) increases the interfacial shear strength (IFSS) between the fiber and the matrix which increments the locking of fiber and matrix notably [14]. Alkalization of hemp fiber increases the roughness of the fiber surface which increases the interfacial bonding property between the fiber and the polymer matrix. But the alkalization process can degrade the cellulosic particles of the hemp fiber which can be considered a challenge as well [69].

Chemical modification techniques like acetylation, treatment with silane coupling agents, grafting copolymerization, and isocyanates-based treatment of the composite polymer can improve the performance of the polymer composite adequately [46]. Acetylation is an esterification process that increases the surface roughness of the composite material as a result better adhesion can be observed [30]. Saline treatment is another acceptable procedure of chemical modification. In this process, a composite of nonwoven hemp fiber –reinforced in unsaturated polyester resin base matrix was undergo the saline treatment process and improved mechanical and thermal properties were found according to the analysis of Panaitescu et al. [75]. Treating polymer composite with polymethylene-polyphenyl isocyanates (PMPPIC) can also modify the fiber and matrix surface but it is not recommended due to the toxic nature of PMPPIC [54].

HFRP has significant scope in the area of polymer composite. Good reinforcement properties, easy availability, favorable costing issues, and notable positive impact on environmental sustainability make this a potential alternative to synthetic composite materials. Though there have been some challenges, diversified modification techniques can improve the constraints notably. In this chapter, the basics of HFRP along with its advantages and challenges will be described thoroughly with appropriate explanation. The chapter will be ended up by providing some future direction with a view to facilitate the manufacturing and application process of HFRP composites.

2 Hemp Fiber Reinforced Polymer Composite

Fiber reinforced polymer composites (FRPCs) are the reinforced polymer materials where the polymer functions as a matrix resin that reaches the bundles of reinforcement fibers and bonds to it. Compared to the synthetic fibers such as glass and carbon fiber, now a days, natural fibers including hemp, jute, flax, and kenaf, are more frequently utilized in FRPCs due to their eco-friendliness, biodegradability, suitable mechanical characteristics, improved energy recovery, and ease of availability. At least two components make up a composite: the matrix and the fibers with distinct physical or chemical properties. A new material with different properties is produced when they are mixed together at a certain ratio following some standard fabrication techniques. The primary purpose of fibers in the FRPCs is reinforcing the polymer plastics. On the other hand, the matrix holds the fiber reinforcements together, distributes loads among the fibers, shapes the net of the composite component, and controls the surface quality.

2.1 Hemp Fiber

For composite parts, high performance fibers such as glass, carbon, or aramid fibers are the most popular options for their enhanced multifunctional properties. Other than these, plant—based natural fibers are also used. The selection of fibers used for a particular application depends on cost, required strength, stiffness, corrosion resistance and other properties for that application. Hemp, flax, jute, kenaf, and ramie are examples of bast fibers among natural fibers. The availability, higher strength-to-weight ratio, production simplicity, and low cost of manufacturing make these fibers ideal for FRPCs. Hemp fibers are considered as one of the strong members of bast natural fibers family, which are derived from the hemp plant under the species of *Cannabis*. Figure 1 shows the morphological structure of hemp fibers [44].

2.1.1 Cultivation and Fiber Extraction

Hemp, the second largest grown bast fiber after jute [28], has gained considerable attention as a reinforcement in the polymer matrices in the last decade due to its renewable, biodegradable, and recycling properties. It was first cultivated in Western Asia. It has several excellent qualities like the ability to grow without the use of pesticides and herbicides and the resistance to fungus, different kinds of weeds, and rodents [47]. Hemp can be grown all over the world [111]; now, hemp is grown in more than 30 nations [84]. Canada, North Korea, and France are the three main hemp-producing nations, with respective production areas of 555,853 ha, 21,247 ha, and 12,900 ha [40]. The soil should have a pH level of 6.0 to 7.5 [9], be between 16–27 °C, and have an adequate amount of moisture [4]. Hemp production is influenced

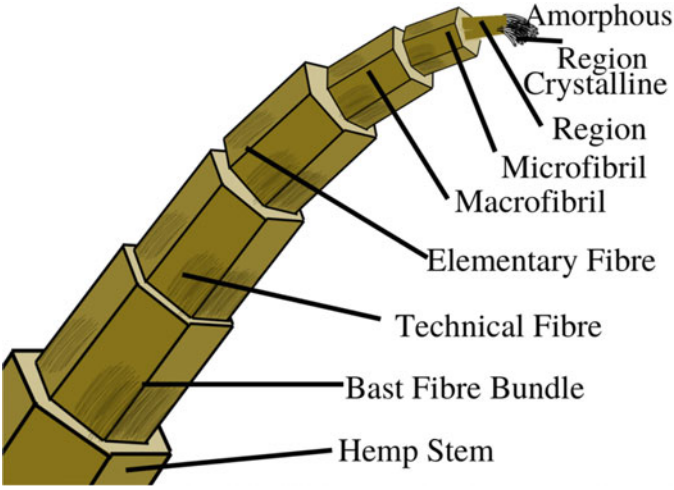

Fig. 1 Morphological structure of hemp fibers

by seeding density, harvest period, and nitrogen content. In one week, the hemp plant can grow 31 cm [74]. Hemp is a tall plant with a short cropping period (70–90 days) [3, 58, 104] and minimal labor-intensive workloads [23]. As hemp has a lesser chance of crop failure than flax, farmers prefer to produce hemp. Additionally, the yield of the hemp-growing area (per hectare) is up to 12 tons for the manufacture of cellulose, 20 tons to produce stems, and 25 tons to produce fiber matter [104]. To obtain fiber that is high-quality in terms of maturity, fineness, strength, and color, producers can adhere to organic cultivation guidelines [18]. Hemp fiber costs 77.63% less to grow than cotton fiber in terms of field preparation, irrigation, seeds, and fertilization [32].

Hemp undergoes a four-step extraction procedure after harvesting. The hemps undergo retting first. Water retting takes 1 to 2 weeks, whereas field retting takes 4 to 6 weeks [3]. During retting, naturally occurring bacteria and fungus break down the pectins and bind the fibers. Using flutted rollers, the second step involves breaking the stems. A beater separates the stems by scutching process after which the fibers are separated and straightened by the hackling process [51].

Cellulose constitutes the majority of hemp fiber (60–70%), followed by hemicellulose (15–20%), lignin (2–4%), pectins (2–4%), and fat (1–2%) [40]. It differs from other natural fibers in many ways, including with its aseptic qualities, excellent absorbency, UV protection, and lack of allergic effects [50]. Table 1 depicts the typical mechanical and physical properties of hemp fiber [12, 37, 98].

2.1.2 Technical Feasibility for Composites

Compared to the majority of the other bast fibers, the lignin percentage of hemp fiber is lower which facilitates the hemp extraction process with minimal fiber wastage [5].

Table 1 Typical mechanical and physical properties of hemp fibers

Properties	Values
Length	8.3–14 mm
Diameter	17–23 µm
Length to diameter ratio	549
Specific gravity	1.5 g/mm^3
Moisture absorption	9.40%
Moisture content	12%
Cellulose content	90%
Water absorption	85%
Tensile strength	310–750 Mpa
Specific tensile strength	210–510 Mpa
Modulus of elasticity	34 Gpa
Microfibril angle	6.2
Elongation at break	2–4%
Torque	62 d/m

Due to the morphological structure of the fiber, fiber processing can be done easily with a descending amount of chemicals [71]. Consequently, the fiber damage rate of hemp compared to the other bast fibers is also minimal. To use as a reinforcing material, fiber strength should be a significant parameter of consideration. Hemp fiber has a satisfactory level of fiber strength for use as a reinforcing material in composites. Better stiffness of the fiber is another advantageous issue of hemp [100]. Though hemp is an annual crop, the cultivation timeframe for hemp is lower than widely used fibers like cotton and jute which make hemp cultivation affordable [5]. Biodegradability and longtime durability are considered the notable assertive impact of hemp [90]. Hemp fibers contribute to reducing the amount of carbon dioxide in the environment [109]. Hereafter, hemp fibers have long wear and tear properties for which the reinforced products are durable. The biodegradability of hemp makes it a potential material for the polymer composite sector. Hemp fibers have some disadvantages as well. The hydrophilic property of hemp disrupts the interfacial bond formation between fiber and polymer matrix. Inadequate bond formation creates cracks in the composites and makes the composite microbial-prone [98]. Though the short cultivation period facilitates the farmers to grow interested in hemp, the storage and transportation capacity of hemp fiber is difficult due to their bulkier hemp stalks. As an annual crop hemp requires a longer timeframe for storage which can create discoloration of the fiber [1]. The mechanical properties of hemp fiber vary according to diversified factors related to fiber cultivation. This is considered another fact of concern while working with hemp fibers [100]. However, the effect of the constraints can be overcome or minimized by applying diversified modification techniques. Being a sustainable alternative to fiber in the composite industry hemp can get significant consideration in the coming days.

2.2 Polymer Matrices

Implementing an effective polymerization technique is elementary to gain the required outcome from the composite material to create bonding among the reinforcement and resin-based matrix material. A Covalent bond is usually formed between the smallest representative units of the substances by which the polymerization process is carried out [10]. Addition polymerization [62], polymerization through free radical formation [24], ionization [11], the ring-opening mechanism [72], condensation [22] are the mentionable techniques which are widely used to execute the polymerization process. Principally, two varieties of polymers are found in the chemical industry synthetic polymers and biopolymers (also known as natural polymers) [20].

2.2.1 Synthetic Polymers

Synthetic polymers are the traditional and most used type of polymer, usually derived from petroleum-based sources. Elastomers, adhesives, and plastics are considered the significant section of synthetic polymers [33]. Tissue engineering, scaffolding, biomedical, hydrogel preparation etc. are the notable areas of polymers where synthetic polymers persist their dominance [39]. Factors like favorable mechanical, thermal and chemical properties make synthetic polymers a considerable choice in the composite industry. The simple and diversified fabrication technique is another vital advantage of synthetic polymers. Due to the beneficiary fabrication technique, this type of polymer can also be used in the packaging sector. Advantageous chemical bonding and interlinking property among multiple polymers are considered another decisive factor for synthetic polymers. Adequate interlinking between polymer matrices is essential to achieve the composite's desired outcome, which can be easily acquired using synthetic-based polymer materials [31]. This bonding property of synthetic polymers can be considered an affirmative factor for tissue engineering purposes. While used in composite material, synthetic polymers can create better interfacial bonding due to their hydrophobicity [83]. However, synthetic polymers also have some mentionable disadvantages. The polymers are made from non-degradable oil-based sources, so they are not environmentally friendly. Some synthetic polymers remain in the environment for decades. As a result, landfill damage and marine environment pollution disrupt ecological balance and environmental sustainability [65].

Along with unsafe disposal, synthetic polymers have toxicity issues as well. Synthetic polymers are obtained by synthesizing various chemical-based products, which may occasionally show toxic behavior. The polymer material can show toxic natural while degradation and link formation in some cases, making them a non-preferable option in diversified medical-based applications and hydrogel formation [20]. Furthermore, the manufacturing of synthetic-based composite materials is not economically friendly. The raw materials of synthetic-based composites are costlier than the natural ones as they are not widely available and efficiently process able. In addition, the manufacturing and processing of the materials have to undergo numerous operations, which can add additional costs while working with synthetic-based composites [19].

2.2.2 Bio Polymers

Biopolymers are a sustainable alternative to petroleum-based synthetic polymers. The bio-degradability properties, recycling, and reusing properties of biopolymers are significantly notable [13]. Biological natural sources like plants, microorganisms, etc., are the prior origin of biopolymers. Pectin, polysaccharide, starch, chitin, gelatin, soybean, gluten, etc., are known widely as protein and carbohydrate-based biopolymers, which are termed agro-polymer in combination [41]. Though, biopolymers are considered incapacitated compared to synthetic polymers while subjecting the mechanical properties of the polymer. However, some biopolymers show significant assertive consequences in diversified mechanical properties. The hydrophilic tendency of natural materials is considered a mentionable constraint that creates hurdles during polymerization due to the deficiency in interfacial bonding [91]. Soybean, an oil-based biopolymer, ensures an effective polymerization process that is entirely moisture-free. As a result, polymers based on dried soybean removes the possibility of deficient bond formation [7]. Due to the adequate thermoplastic property, starch gets attention as a biopolymer material despite being a polysaccharide-based natural substance [25].

By incorporating polycaprolactone with starch-entrenched plastic materials, an effective biopolymer named Mater-Bi® is formed as polycaprolactone is a variety of recycled polyester, so the output material shows its excellence in physical and mechanical properties like synthetic polymers but eliminates the drastic environmental impact of the synthetic polymers [64]. In addition, chitin is another predominantly mentionable biopolymer for its rigidity and variable usability, while the biopolymer source is polysaccharides derived from microorganisms [88]. Polyester base materials with biodegradability, like polycaprolactone (PCL), are also considered biopolymers due to their natural source of polymer formation.

Usually, while considering diversified properties, biopolymers require to catch up with the parameters compared to most synthetic polymers. While mechanical strength, stiffness, etc. properties are the key point of synthetic polymers, their unsustainable disposal can consider as the major weakness of the mentioned polymers. However, natural biopolymers are rising towards technical feasibility by implementing diversified surface modification techniques according to the base materials' requirements. The absence of toxicity in the biopolymers is another notable impact that enriches the application of biopolymers in sectors like hydrogel formation, scaffolding, medical textiles preparation, etc. Furthermore, compared to the raw materials of synthetic polymers, biopolymers are economically friendly with easy availability.

Consequently, the processing with biopolymers is affordable compared to synthetic ones. However, there is also some adverse impact of using biopolymers, like the chances of microbial attack on the polymer surface as they are not resistant to microorganisms. Biopolymers' leisurely processing and manufacturing create the possibility of wasting time and energy. As the environmental and atmospheric condition varies while maintaining the process parameters of biological polymers, acquiring identical output for every batch is challenging. The possibility of heavy metal contamination in plant-based biopolymers can also be regarded as a constraint while processing [20]. Sustainability and environmental protection for the coming days are considered essential issues on the globe. So, to acquire effectual output by harming the world minimally, replacing the biopolymers with synthetic ones is very important. Though biopolymers also have some constraints, most can be minimized or removed by applying effective modification techniques.

3 HFRPs Fabrication Techniques

Fabrication of hemp fiber reinforcement polymer composites (HFRPs) involves preparing hemp fiber preforms (such as woven, knitted, braided, and long sheets or mat structures of fibers) and then reinforcing with polymer matrix by different techniques. HFRPs fabrication techniques can be divided into two general classes: open and closed molding techniques based on the mold types used while making composites. In the open mold process, the fiber-matrix are placed in an open mold and cured in an open surface, whereas the fiber-matrix are placed and cured in a closed surface in the case of closed mold process. Composite fabrication techniques can be classified based on many factors as shown in Fig. 2. However, the most common HFRPS fabrication techniques are discussed as follows.

3.1 Hand Lay-Up Technique

The hand lay-up technique is depicted in Fig. 3 [108] which is used widely to fabricate the HFRPs due to its simplicity. The processes involved with this technique include mold preparation/gel coating, lay-up, and finally curing. Below is a description of each step:

I. Mold preparation/Gel coating: To facilitate easy removal and prevent sticking material to the surface, the mold wall is coated with an antiadhesive substance (i.e., wax). Additionally, to provide a smooth surface for the finished product, a thin sheet of plastic is preserved at the top and bottom of the mold plate. A pigmented gel coat can also treat to obtain a light shade of the product.
II. Lay-up: The layers of fibers in the shapes of woven, knitted, stitched, chopped, etc. are placed onto the surface of the mold by hand until the desired thickness is reached. If necessary, the staked layers are cut into the required shape. The resin substance (epoxy, vinyl ester, unsaturated polyester, etc.) is then carefully

Fig. 2 Frequently used hemp fibers reinforced polymer composite fabrication techniques

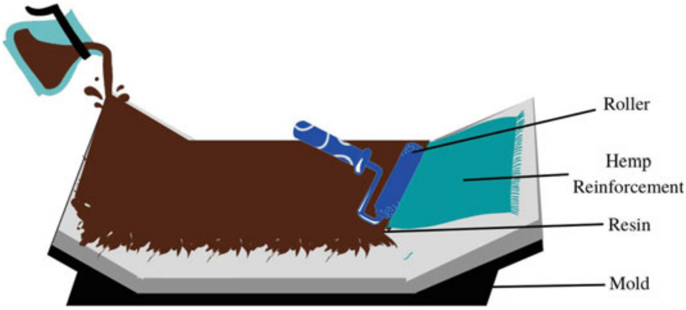

Fig. 3 The schematic diagram showing hand lay-up technique

poured or applied to prevent the creation of air bubbles, and it is dispersed evenly using a brush. The resin is then forced into the layers with a trowel or hand roller to provide enhanced contact between the matrix and the fiber reinforcement and to provide a uniform distribution. After that, the mold is sealed, and the pressure is let out.

III. Curing: Curing is the hardening of HFRPS without any external heat, usually in standard atmospheric conditions. After curing, the mold seal is opened, and the composite is withdrawn from the mold surface.

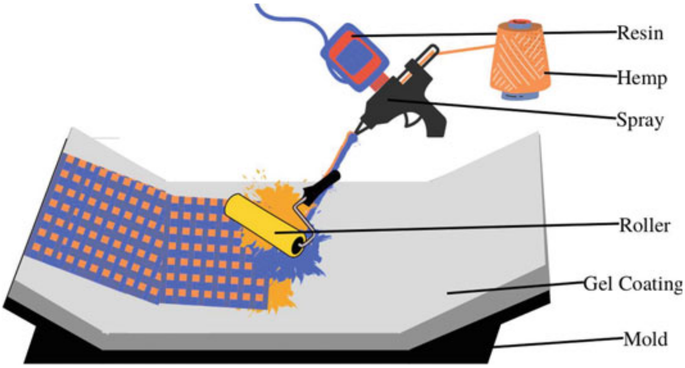

Fig. 4 The schematic diagram of spray-up technique

3.2 Spray-Up Technique

Spray-up technique is like hand lay-up technique excepting a handgun is used to spray resin and fibers on a mold. Usually, the spray gun uses compressed air to chop the fibers and spray them alongside the glue. Simultaneously, a hand roller fuses the chopped fibers into the resin and eliminates the trapped air between the layers. Finally, curing is done in standard atmospheric conditions like hand lay-up technique. The spray-up fabrication technique of HFRPs is illustrated in Fig. 4 [86].

3.3 Filament Winding Technique

Filament winding technique is used to produce the HFRP composites that are cylindrical in shape. In this technique, a barrel-shaped rotating mold named as 'Mandrel' is used. Usually, fiber roving are pulled and passed through a resin bath and wound around a pivoted mandrel as shown in Fig. 5 [86]. There are basically three different types of winding patterns in this technique, i.e., helical, polar, and hoop windings. After winding required layers with desired shape of the product, curing is done in standard atmospheric conditions. Recently, the filament winding technique is developed which is equipped with an industrial robot for self-automation.

3.4 Pultrusion Technique

In pultrusion technique, the fiber roving is first passed through a bath and then through a series of heated dies and pulled by a puller simultaneously. The bath contains matrix materials (i.e., poly(butylene succinate) mixed with organoclay which are

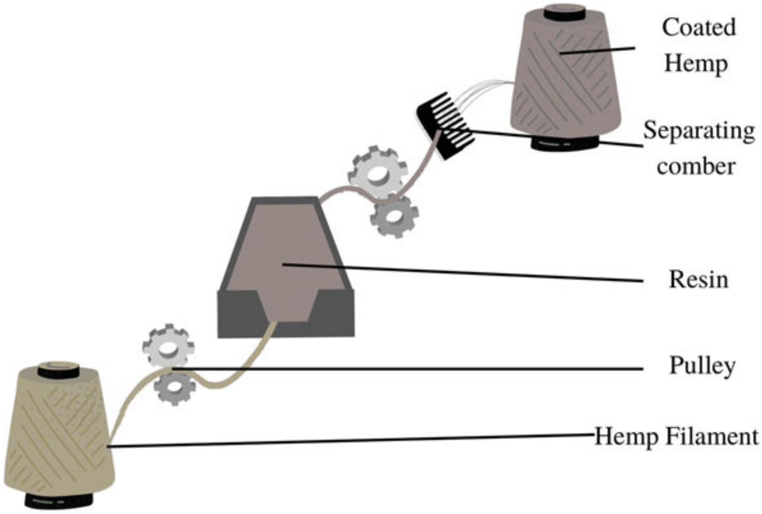

Fig. 5 The schematic diagram of filament winding technique

extruded from a single-screw extruder as shown in Fig. 6 [53]. The final product can be rectangular, circular, square, H-shaped and I-shaped by following the die cross-section. After that, composite materials are cured in one of the dies and cut to a required length.

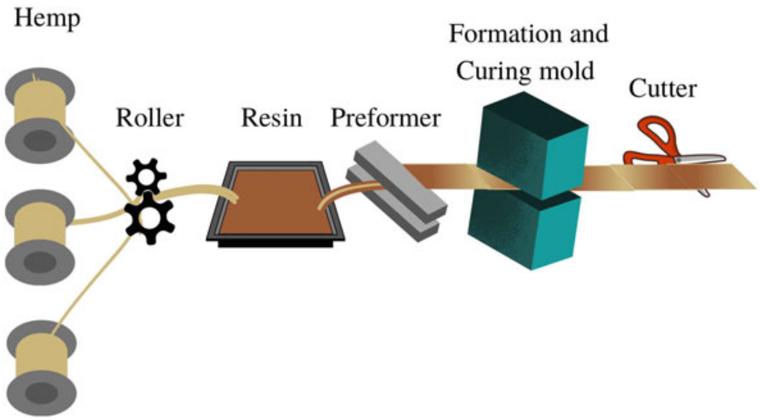

Fig. 6 The schematic diagram of pultrusion technique

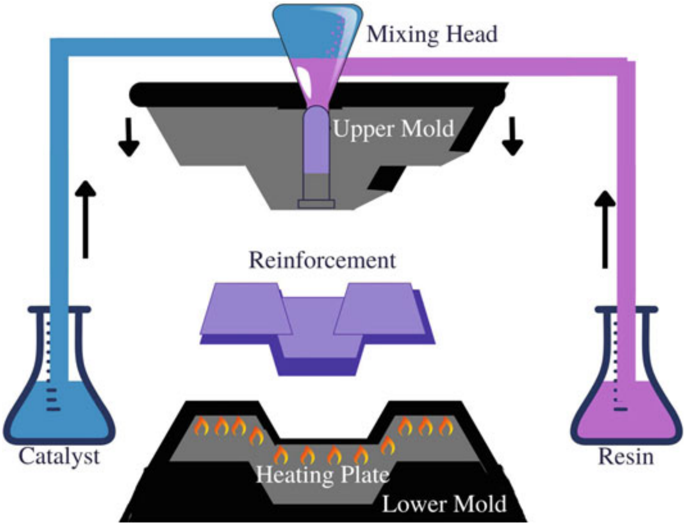

Fig. 7 The schematic diagram of RTM technique

3.5 Resin Transfer Molding (RTM) Technique

In RTM, hemp fiber either woven roving or mat are cut using a knife or scissors and a template into a preform. The preform mat or roving are bound with thermoplastic binder and then arranged inside the lower half of the mold cavity. The preheated resin is pumped with a high pressure and transferred into the mold cavity through an injector. Resins usually applied in RTM include polyester, epoxy, vinyl ester, and phenolic. The working principle of the RTM technique is shown in the Fig. 7 [27].

3.6 Vacuum Assisted Resin Transfer Molding (VARTM) Technique

In vacuum infusion or VARTM technique, preform fibers are kept on a mold and a perforated film is placed between vacuum bag and resin container. The resin is sucked by the vacuum force through the perforated films. After the impregnation, curing is done at room temperature. The difference between VARTM to RTM, the replacement of the top portion of a mold with a vacuum bag to enable resin flow.

3.7 Vacuum Bagging Technique

Figure 8 shows the vacuum bagging process for HFRP composites. Vacuum bagging or vacuum bag molding is an advance version of hand lay-up technique. In this technique, firstly, prepreg laminates are placed onto the foundation of horizontal mold using the same hand lay-up technique. Secondly, a series of equipment and materials (i.e., layer of release film, air-bleeder, blocked film, and breather) are stacked in layers. Thirdly, a vacuum bag (i.e., polythene sheet) is used to cover the composite laminate and all the layers. Finally, a sealer is used to seal the vacuum bag and a vacuum pump is used to draw out the air between the vacuum bag and mold.

3.8 Compression Molding Technique

Figure 8 [105] shows the overall process of compression molding to fabricate HFRP composite. In this technique, first, hemp fibers and polymer matrix are fed into a twin extruder and an intermediate material called molding compound is prepared. Second, the compound material is extruded through a rectangular shaped die to form a tape. Third, the compounded tape is cooled, chopped into several pieces and put into a low shear plasticator. Fourth, the shear plasticator is charged and placed into a mold and the compression molded into a plate at a required pressure. Finally, the compression molded plate is demolded after a while.

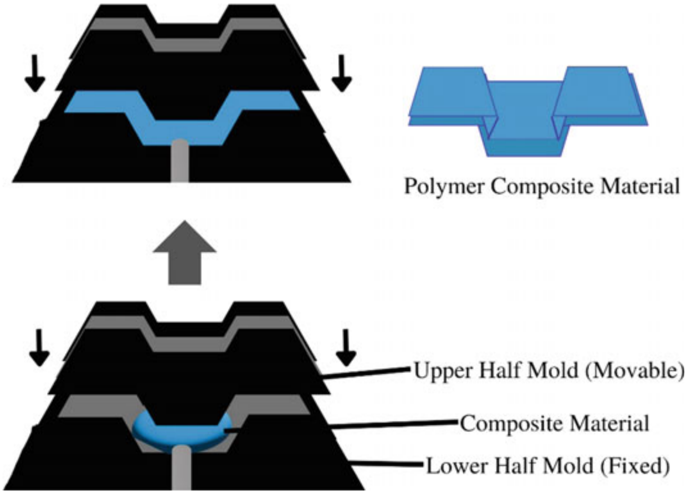

Fig. 8 Compression molding technique of HFRP composites

3.9 Injection Molding Technique

In injection molding technique, at first the molding compound is prepared in the form of granules or pellets by using a twin-screw type of extruder. Then, the molding compound is fed into the mold through a hopper and a barrel type heater, as shown in Fig. 9 [86]. After the action of screw and heated barrel, molding compound is melted. Finally, the molten compound is injected into the mold through a nozzle to form the HFRPS. After that the composite part is cooled and it acquired the definite shape.

4 Factors Affecting Interfacial Bonding

Interfacial bonding can be defined as the bond or interaction between polymer matrix and the reinforcement materials. Mechanical properties of fiber reinforced polymer composites largely rely on this interfacial adhesion [76]. Various aspects of fiber can influence interfacial adhesion, such as fiber amount, alignment, length, and fiber construction. Additionally, the fiber's dispersion consistency in the matrix, composite construction techniques, and various fiber treatments affect the interfacial adhesion. Thus, the mechanical properties of the reinforced polymer composite can be affected.

The polymer composites can achieve improved mechanical properties by using a specific portion of natural fibers. After exceeding this certain limit, the properties start deteriorating. Crossing a certain point makes the polymer matrices unable to reach every fiber, which leads to defective fiber wetting. Therefore, a reduction in transferred load occurs in the fiber-matrix interface region. Besides, the number of voids increases in the composite. A high number of voids creates stress concentration resulting in declined mechanical properties.

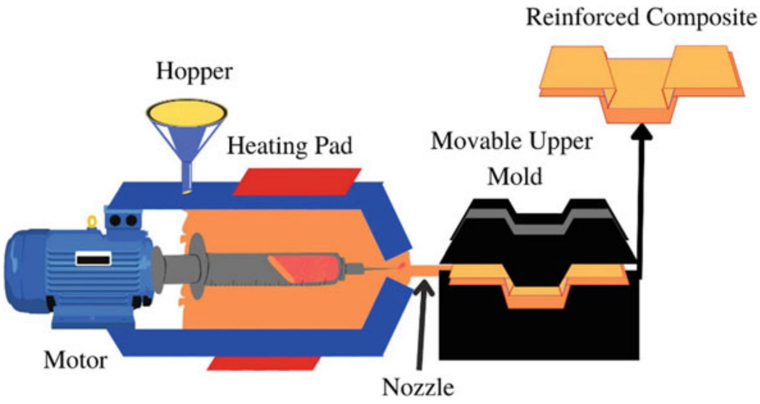

Fig. 9 The schematic diagram of injection molding technique

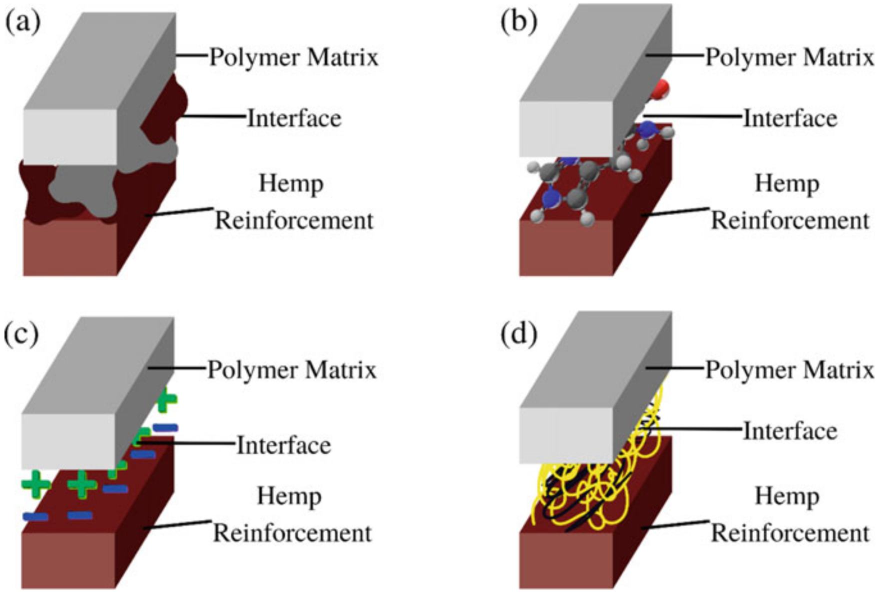

Fig. 10 Schematic diagram of interfacial adhesion between matrix-fiber by **a** mechanical interlocking, **b** chemical bonding, **c** electrostatic adhesion, **d** intermolecular interaction

Fiber alignment has a significant role in controlling the mechanical properties during the reinforcement of polymer composites. In the case of a composite, the higher fiber alignment from the axis makes the mechanical properties worse. If the external load performs at a significant angle regarding the axis of the primary fiber, the composites experience a decrease in their mechanical properties. Furthermore, biopolymers and natural fibers influence polymer composite properties. Nevertheless, fiber is more responsible for the mechanical characteristics of the composite than polymers. Moreover, mechanical properties can change with pressure, temperature, and time during various processing [41].

As shown in Fig. 10 [41], interfacial interaction between fibers and matrix is achieved by any one of the following mechanisms.

- Mechanical interlocking,
- Chemical bonding
- Electrostatic adhesion
- Intermolecular interactions (Hydrogen bond and Van der Waals force).

4.1 *Mechanical Interlocking*

Interfacial bonding can be created by interlocking the natural fiber's rough surfaces with polymer matrices. A schematic diagram of interfacial adhesion between matrix

-fiber by mechanical interlocking is shown in Fig. 10a. Relying on the intensity of fiber surface roughness, the area bound between the fiber and matrix will change proportionally. Accordingly, the interfacial bonding strength varies. Mechanical interlocking in natural fiber polymer composites is affected by waxes and fats present in the fibers, which are undesirable for enhanced interfacial interlocking.

The difference in the thermal contraction or extension between the matrix and the fiber causes stress of clamping. This incident helps mechanical interlocking to ensure improved interfacial bonding. Mechanical interlocking depends on the direction and the properties of the surface. More interlocking can be achieved by augmenting the roughness of the fiber surface which causes better interaction with the polymer matrices. As the interlocking between fibers and polymer matrix increases, the mechanical properties of polymer composites increase [63].

4.2 Chemical Bonding

Fibers and polymer matrices can be bonded through different types of chemical bonds, such as covalent, ionic, and metallic bonds. In the case of natural fiber-reinforced polymer composites, covalent bonds are more available than any other bonds. Natural fiber's hydroxyl groups and the polymer resin's functional groups share electrons. Thus, covalent bonding forms between the matrix and the fiber. A schematic diagram of interfacial adhesion between matrix-fiber by chemical bonding is shown in Fig. 10b. Interfacial adhesion can be affected positively by adding coupling agents with the functional groups while creating these covalent chemical bonds.

4.3 Electrostatic Adhesion

When the natural fibers and the polymer matrix come into close contact and undergo friction, electrical charges are transferred between them. Since the polymer is an insulator, the electric charge cannot move and stays there. This charged polymer matrix, when brought close to the fibers, introduces equal and opposite charges to the surface of the fibers. The law of 'opposite charges attract each other' comes into play and the polymers are attached to the fibers. A schematic diagram of interfacial adhesion between matrix -fiber by electrostatic adhesion is shown in Fig. 10c. The strength of the interfacial bonding depends on the density of those charges, caused by this electrostatic adhesion. When the composite's components are detached, the attraction resulting from electrostatic adhesion becomes effective [63]. Nonetheless, the impact of electrostatic adhesion in interfacial bonding is less than the Van der Waals forces since it is pH, temperature and humidity sensitive [46].

4.4 Intermolecular Interaction

Hydrogen bonding or Van der Waals forces cause close intermolecular interaction between the molecules of the matrix and the fiber which results in interdiffusion. The matrix and the fiber interacted closely by spreading and penetrating in the absorption stage which is the first stage of this type of adhesion. After this, molecular attractions like Van der Waals create adhesion [15]. Appropriate wetness creates interdiffusion between the matrix and the fiber molecules. A schematic diagram of interfacial adhesion between matrix -fiber by intermolecular interaction is shown in Fig. 10d. Ability of the penetration of the substrate and the chemical competency of the two components affect the degree of this interdiffusion. In spite of these advantages, this diffusion can impact the interfacial bonding by debonding of fiber, bridging of fiber etc. in a certain atmospheric exposure. In addition, the diffused molecules may get intertwined with remaining molecules. This intertwining can be affected by temperature. With the increase of the temperature, the molecules attain molecular motion by overcoming hindrances. When the temperature reaches the glass transition temperature, molecular intertwining fails to prevent slipping of the molecule chain. This incident results in the weakening of interfacial bonding as molecular intertwining gets steadily unraveled [63].

5 Effects of Interfacial Bonding on Fatigue Behavior

Damage mechanism is an instrumental factor of the fatigue behavior of NFRPs, as the degradation of the composite takes numerous cycles before final rupture. Thus, improvement of the fatigue characteristics of HRPCs can be achieved by delaying the fracture mechanisms or improving the key factors contributing the fracture. Several cutting edge techniques are devised and applied to analyze the damage mechanism such as acoustic emission [29], thermal imaging and micro-CT analysis [16] etc. For morphological analysis, SEM, TEM and microscopic analysis are generally employed to observe the fractured surface [101].

The hydrophilic nature of NFs is a major contributor of NRFPCs weak fiber-matrix adhesion. Alkaline treatment turns the hydroxyl group present in NFs to alkoxide, which leads the NF to become less hydrophilic and forms a rougher surface [34]. Chemical treatment also increases the fibrillation of NFs, therefore enlarging the effective surface area [2]. Fiber volume fraction dictates the failure mechanism to a great extent. Matrix related failure like cracking of matrix normal to stress, brittle fracture generally occurs in composites with lower fiber percentage. On the contrary, fiber dominated fracture mechanism like delamination of layers, fiber breakage, splitting occurs in composites with high fiber volume fraction [97]. The T-T stress ratio contributes to brittle fracture along with fiber delamination, splitting and breakage as well as fracture of matrix, which is contributed by mode-I and mode-II type fracture mechanism described by opening and in-plane shear mode respectively. The T-C

Fig. 11 Failure modes of HRFCs for **a** T-T, **b** T-C and **c** C–C stress ratio; reused with permission from [97]

and C–C stress ratios display mainly mode-II type failure demonstrated by kink type fracture mechanism. Figure 11 depicts all types of fracture mechanism pertaining to fatigue failure.

Acoustic emission provides another interesting aspect about the failure mechanism for environmentally conditioned composites. For low stress level, the AE events pertaining to damage for the composites at reference condition starts after about 20% of the life cycle, whereas for the 90 days wetted in water composites the significant AE events appear almost at the beginning of the fatigue cycles. However, the dried after wetting composites portray different characteristics, the damage appear much faster than the reference condition composites. It may be attributed to the residual stress development because of the variation of drying temperature and time of various components of composites [16]. The initial matrix damage turns to fiber-matrix interface over the fatigue cycle in ambient conditioned composites, whereas the matrix fracture and interfacial debonding occur simultaneously in wet condition from the beginning. In case of wet/dry composites, both the failure mechanism occur gradually. Quantitative analysis using micro-CT displays variation in damage of wet and wet/dry composites in comparison with reference ones. Moreover, micro-CT analysis reveals the initiation and propagation of fracture elaborately. The initiation of fracture occurs at the fiber-matrix interface, and propagates through the interfacial boundary from, one to another as shown in Fig. 12. Thus, several fiber-matrix debonding contribute a final crack across the laminate thickness. IR imaging process provides quantitative value of heating and temperature of the composite sample. The heating of sample generally happens due to the crack initiation or propagation or friction among the constituent materials. Although, extracting any information pertaining to the fracture mechanism is yet to be researched [29].

Alkalization of NFRPs yield better interfacial bonding while using a certain percentage of alkaline solution. Treatment with up to 5% alkaline solution successfully improved the fatigue characteristics by improving the fiber-matrix interfacial bonding and higher resistance to water absorption, which in turn increase the interfacial bonding further [101].

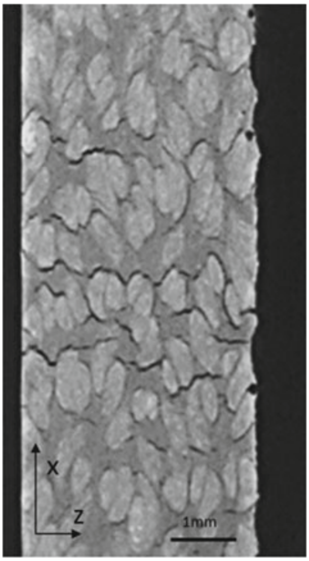

Fig. 12 Micro-CT analysis of wet/dry conditioned composite showing the fracture initiation and propagation; reused with permission from [16]

6 Techniques to Improve Interfacial Bonding

In HFRPs, the major components are the reinforcement hemp fiber and the polymer matrix, and the qualities and performance depend on their interfacial bonding. The typical thermosetting polymer matrices are non-polar (hydrophobic) substances incompatible with polar (hydrophilic) hemp fibers. They, therefore, cause poor adhesion between the fiber and matrix interaction surfaces. So, modifications are needed either in fiber or matrix or in both to enhance the interfacial bonding. There are developed several techniques to improve the hemp fiber-polymer matrix interfacial bonding which is depicted in Fig. 13 and discussed.

6.1 Fiber Modification Techniques

In HFRP composites, the hydrophilic character of hemp due to strongly polarized cellulose makes it incompatible with the hydrophobic non-polarized polymer matrix. Besides, the poor fiber-matrix interfacial bonding arises from the natural wax present in the cuticle of the fiber which limits wettability and adhesion to the synthetic matrix. So, fiber modification is needed for strong interfacial bonding between fiber and matrix. Therefore, hemp has to be specially modified to effectively transfer stress from matrix to hemp to achieve moisture repellence, flame retardant, bio-durability, weatherability or other properties. Various methods have been developed for fiber modification as shown in Fig. 13 which will be described in this section.

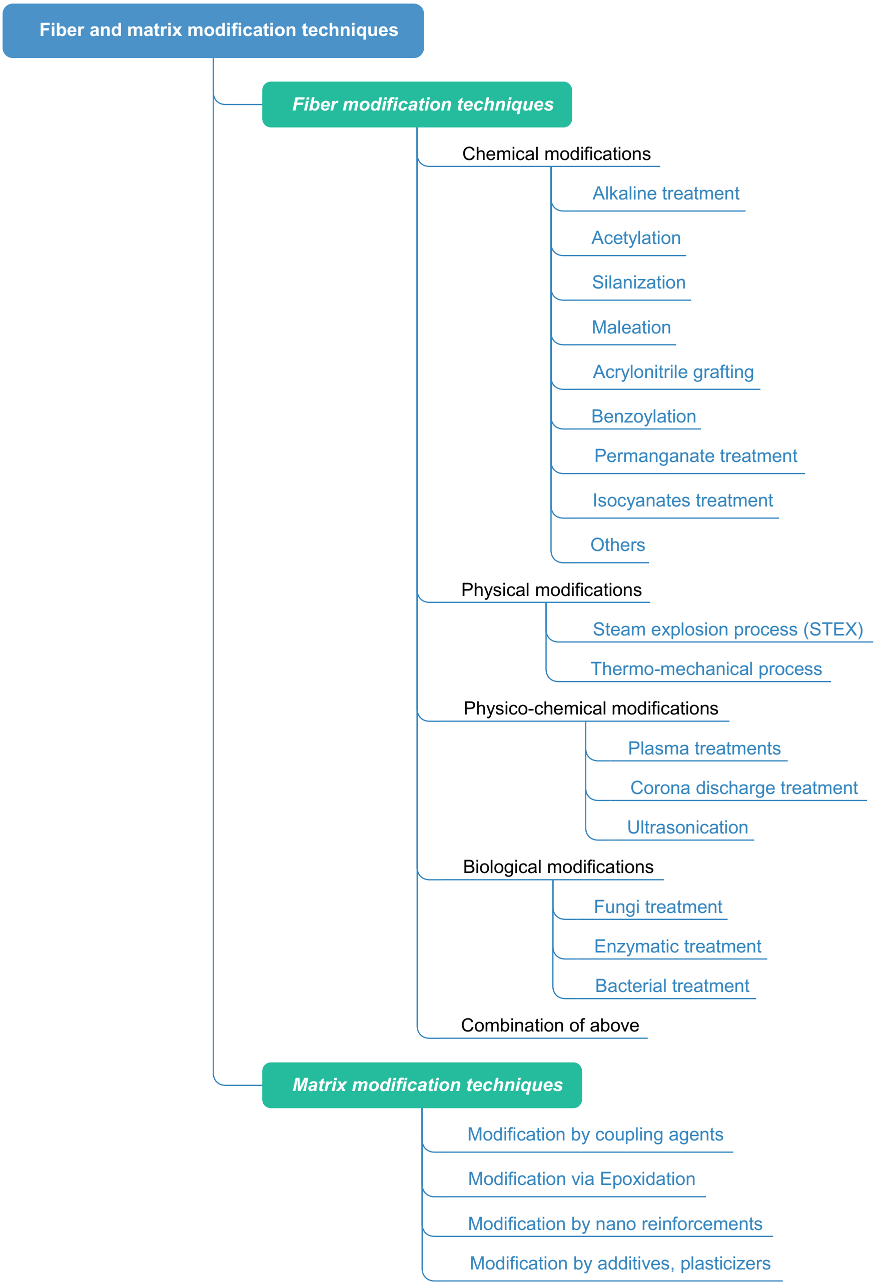

Fig. 13 Different techniques to improve interfacial bonding between hemp fiber and polymer matrix

6.1.1 Chemical Modification

Hemp fibers are chemically modified to reduce the hydrophilic character of it and the crystalline index of cellulose as well as to enhance the interfacial bonding, mechanical properties, dimensional stability, and improved performance of hemp fiber with polymer matrix in composites. Various chemical treatments are used for fiber modification following the application envisaged, production costs, and environmental impact. The most commonly used chemical treatments for hemp fibers are alkaline treatment, acetylation, benzoylation, acrylation, acrylonitrile grafting, permanganate treatment, isocyanate treatment, silane coupling, maleated coupling etc. A short brief of each chemical treatment is given below.

Alkaline Treatment

The alkaline treatment, also known as mercerization of hemp fibers is employed to partially reduce the lignin, pectin and hemicellulose as well as wax and oil. The treatment enables better fiber wettability and a better fiber-matrix adhesion due to a larger effective surface area created by the reduction of impurities. In addition, it causes the cellulose chains to pack better and more closely and increases crystallinity. Consequently, if the fibers are exposed to a high alkali concentration after this alkaline treatment, it may lead to deteriorated fibers and unable to support an efficient load transfer. Sodium hydroxide (NaOH) is commonly employed in alkaline treatment of hemp fibers because it improves surface roughness and makes it possible to modify the fiber further chemically or physically. A typical alkaline reaction of hemp fiber is shown in Fig. 14.

The mechanical properties of the hemp fiber depend on the concentration of the NaOH, temperature, treatment duration, stress level, cellulose amount and the crystallinity index of cellulose. Some findings of the literature are summarized below, showing that the alkaline treatment improves the mechanical properties of hemp fiber:

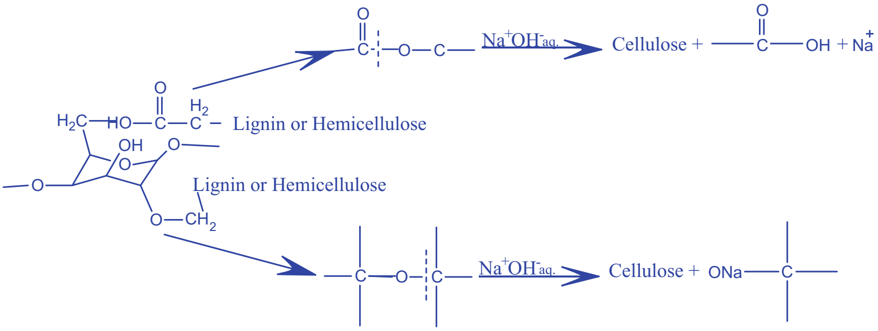

Fig. 14 Elimination of lignin or hemicellulose by hydrolysis of ester and ether bonds of hemp with alkyl treatment

- When the NaOH solution concentration was 6%, the tensile strength peaked at 1050 MPa, while the maximum Young modulus value (65 GPa) was attained at a 4% concentration [68].
- The alkaline treatment raised composites' tensile strength, modulus, and strain by 47%, 22%, and 66%, respectively, compared to composites made with raw hemp fibers [115].
- Greater thermal stability is found when the hemp fibers are treated with 5 wt% NaOH, for 24 h at 50 °C [6].
- It is found that combining an alkali treatment with 12% NaOH solution and high-pressure defibrillation used after cryogenic crushing reduces the diameters of the hemp fibers from micro to nano scale [110].
- The hemp fibers' diameter, tensile strength, and Young's modulus values reached their highest levels after the NaOH-Na_2SO_3 combined treatment, however nanocellulose components are reduced slightly [17].
- After the combined NaOH–Na2SO3 treatment, the fibers are immersed in a bath of vegetable oil (soybean and corn oil) with acetone solution. This enhanced their hydrophobic character, which improved their compatibility with the matrix and increased their mechanical strength. The related composites displayed longer service lifetimes and superior mechanical performance [73].
- At different temperatures (room temperature and boiling point) and with varying alkali solutions' concentrations (2.5%, 5%, 7.5%, 10%, and 15% w/v, respectively) demonstrated the highest sound sorption performance from treatment at higher temperatures and concentrations [113].
- Weaved hemp fibers treated with NaOH, a commercial fire retardant, or a combination of the two chemicals are used to strengthen vinyl ester resin. According to burning tests, thermogravimetric analyses, and limiting oxygen index tests, the treatments improve composites' fire retardant qualities [60].

Acetylation

This treatment involves esterifying part of the hydroxyl groups present in the fiber structure with acetic, propionic, or even vinyl acetate at high temperatures in organic media while the presence of a catalyst (such as sulfuric acid, pyridine, sodium acetate, or potassium). Acetylation is often conducted following the alkaline treatment and frequently used in conjunction with other treatments, such as silanization or the use of compatibilizing reagents, which improve the tensile strength, wettability, interfacial bonding as well as thermal stability of the corresponding composites [21]. The mechanism of hemp fiber esterification with acetic anhydride is shown schematically in Fig. 15.

Silanization

Hemp fibers are silanized by being submerged in a water-and-alcohol-based silane solution that has been diluted. In Fig. 16, a schematic representation of a silane treatment, silanol is further reacted with to form stable covalent bonds (cellulose-O-Si), enhancing the fibers' surface area. Alkali-treated hemp fibers are modified using

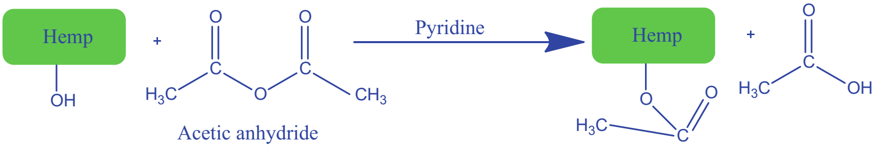

Fig. 15 Esterification of hemp fiber with acetic anhydride

three different silanes as coupling agents. It was revealed that the silane treatment enhanced the tribological characteristics of the corresponding composites. At the highest fiber loading (20 wt.%), silane-treated hemp fiber/cyanate ester and benzoxazine mix resin composites demonstrated considerable improvements in mechanical parameters regarding flexural strength and micro-hardness. Additionally, there was a noticeable increase in thermal stability and glass transition temperature [116].

Maleation

Maleic anhydride (MA) reacts with the hydroxyl groups in the amorphous region of hemp fiber and diminishes to make it hydrophobic. It functions as a compatibilizing agent to enhance the interfacial bonding between fiber and matrix and to improve the respective composites' Young's modulus, flexural modulus, hardness, and impact strength. The chemical reaction of hemp fibers with MA is schematically depicted in Fig. 17. The same outcome is observed when hemp fibers are treated with maleated polyolefins (maleated polypropylene (MAPP) or polyethylene (MAPE)) instead of MA [67, 105].

Acrylonitrile/Methacrylic Anhydride Grafting

Grafting is a successful approach to covalently bind the reinforcement fibers to the macromolecular chains of the polymer matrix. After grafting, the surface energy of the fibers is raised to a level equivalent to that of the matrix, resulting in improved interfacial adhesion and greater wettability of the modified fiber. Figure 18 illustrates how the grafting process generates bridges between hemp and matrix in ether/ester in the presence of acrylonitrile or methacrylic anhydride.

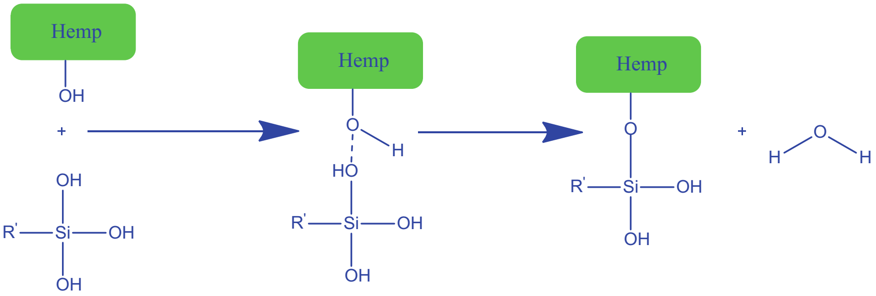

Fig. 16 Silanization of hemp fibers (where R´ represents alkyl radical)

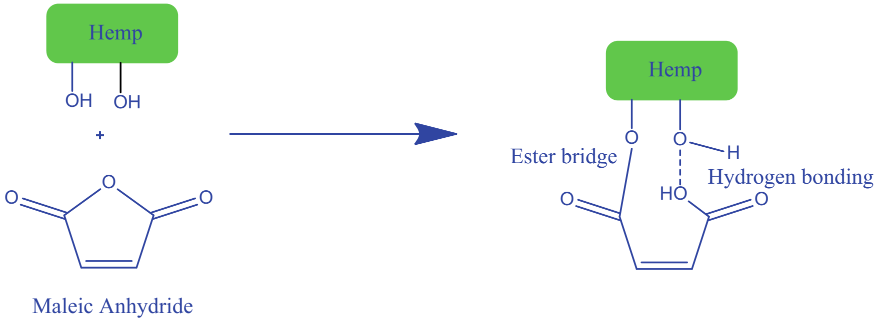

Fig. 17 Hemp fiber maleation using Maleic anhydride (MA)

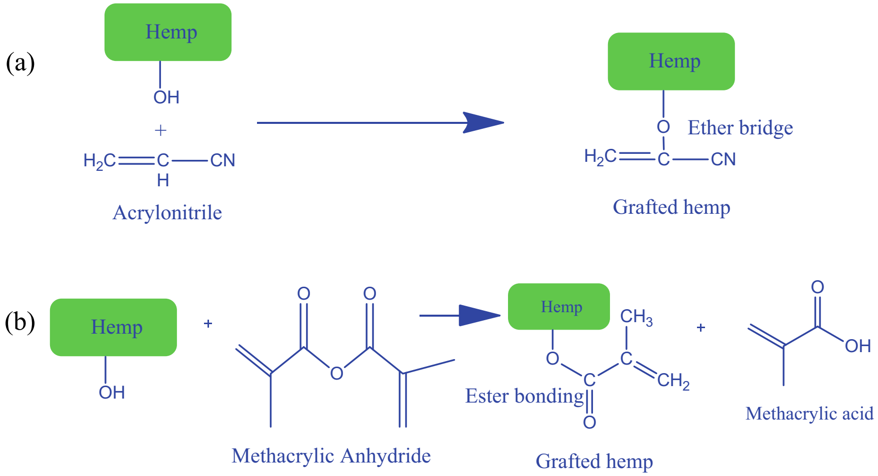

Fig. 18 Grafting copolymerization of hemp fiber with: **a** Acrylonitrile and **b** Methacrylic anhydride

Acrylation occurs when hemp fibers react with acrylic acid and refer to as the grafting reaction in the presence of methacrylic anhydride. The reactive hydroxyl groups in hemp fibers must first undergo an alkaline pretreatment to be easily esterified. Acrylation results in fibers with greater thermal stability and crystallinity [45].

Benzoylation

Benzoylation of hemp fibers is the treatment of hemp with benzoyl chloride as shown in Fig. 19. It greatly increase the hydrophobicity and thermal stability of fibers, which in turn boosts the interfacial adhesion between the fibers and polymer matrix [26].

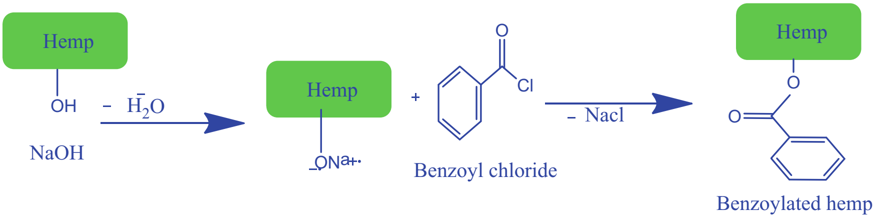

Fig. 19 Benzoylation of hemp fibers

Permanganate Treatment

The hemp fiber-polymer matrix interface's interlocking is improved by grafting using potassium permanganate in acetone. KMnO4 can also remove lignin concurrently by reacting with hydroxylic groups in hemp lignin. It should be noted that if the permanganate solution concentration is more than 1%, treatment leads to degraded fibers [43].

Isocyanates Treatment

Hemp fibers treated with isocyanates show enhanced hydrophobicity and improved interfacial bonding. The reaction of isocyanates with hemp is known as the coupling reaction, where the isocyanate moiety reacts with hydroxyl groups and strengthens the corresponding composite by creating a urethane bond [43].

Other Methods of Chemical Modification

There are also available techniques to modify hemp fiber chemically such as etherification, oxidation, sole-gel, peroxide treatment, sodium chlorite treatment, liquid ammonia treatment, triazine treatment and treatment with stearic acid, salicylic acid, phosphoric acid.

6.1.2 Physical Modification

The physical modifications of hemp fibers are done to separate filaments individually from fiber bundles. Since physical methods do not use poisonous or hazardous reagents or special equipment for chemical synthesis, separation and purification, and the removal of by-products and unreacted compounds, they are cleaner and easier to use than chemical ones. In most cases, physical techniques are employed to separate fibers without changing their chemical components, while enhancing the fibers' compatibility with the matrix by altering their structural and surface characteristics. There are several physical techniques to improve interfacial bonding such as steam explosion, thermo-mechanical treatment, microwave irradiation, milling, ultrasound treatment, stretching, and calendaring. The most common steam explosion process and thermo-mechanical treatment for hemp fiber modification is described below.

Steam Explosion Process

The steam explosion involves heating the ligno-cellulose of hemp fiber and breaking it down mechanically through explosive decompression. At the same time, fiber is chemically modified through the hydrolysis of hemicellulose, modification of the lignin structure, and reduction of the crystallinity index. It separates individual filament from fiber bundle with a smoother surface, higher bending strength and decreased stiffness.

The separation of the lignocellulosic biomass components of hemp fibers can be accomplished using either the uncatalyzed steam explosion (using only steam water) or the catalyzed steam explosion (adding acidic reagents to the steam water). But, the catalyzed steam explosion is currently recognised as one of the most cost-effective pre-treatments because it provides enhanced sugar recovery, fibers with increased susceptibility to enzymatic digestion, usage of lower temperatures [52]. Recently, cottonized hemp fibers are produced by impregnating hemp fibers with an 8% NaOH solution and steam processing them for 4.1 min at 191 °C [92].

Thermo-mechanical Techniques

Thermo-mechanical techniques imply a thermal treatment (Temperature close to glass transition temperature of lignin) to separate single fiber by releasing lignin from fiber bundles of hemp. The thermal treatment of hemp fibers carried out in a neutral environment provide opened fiber without causing any harm [81]. The same significant result is found when hemp fiber is treated thermally in presence of nitrogen instead of treatment in air at a high temperature (over 220 °C) [82].

6.1.3 Physico-chemical Modification

If the fiber modification along with cleaning is needed without changing the chemical composition of fibers then the physico-chemical techniques are employed. The most common physio-chemical modification of hemp fibers are done by plasma treatments, corona discharge treatment, high energy radiation methods and ultrasonication. A short description of each method is discussed below:

Plasma treatments: Plasma treatments with low temperature and under atmospheric pressure are employed to modify hemp fibers. The treatment significantly improves the wettability and dyeability of hemp fibers by creating a rougher fiber surface [14].

Corona discharge treatment: Corona discharge treatment is employed at a low temperature and atmospheric pressure to enhance the wettability, adhesion, and compatibilization of a polymeric surface of a composite with low cost. In the case of PP-based composites, the hemp fibers treated with corona discharge exhibit an improved Young modulus of roughly 30% [85]. Positive outcomes are also obtained when hemp fibers are treated with corona discharges where natural resins are used as matrices [80].

Ultrasonication: This method removes the non-cellulosic components from the alkyl-treated hemp fibers (15 min at 50C; 40 kHz; 100 W), resulting in smooth, clean fibers with a high strength and modulus and improved wettability [103].

6.1.4 Biological Techniques

Biological treatments use low energy, processing conditions, non-toxic chemicals as well as healthy technology while needing a large space, controlled environment as well processes are slow. Hemp fibers can be modified by bacteria, enzymes, or fungi, three different biological techniques. The bacterial technique uses nanotechnology to deposit bacterial cellulose onto hemp fibers, whereas the two earlier procedures primarily employed to modify the surface features by eliminating specific fiber components.

Bacterial treatment: The technique entails growing nanosized cellulosic deposits directly on the surface of hemp fibers while using a cellulose-producing bacterium (such as *Acetobacter xylinum*, *Gluconacetobacter xylinus* strain BPR 2001), regulated culture medium, and environmental conditions. The deposition of bacterial nano-cellulose allows better control of the interfacial contacts between the modified hemp and matrix by exploiting the interphase thickness and properties. Greater fiber wettability results in enhanced interfacial adhesion between the hemp fibers and the polymer matrix through mechanical interlocking [45].

Enzymatic treatment: Although it requires a controlled environment, costly enzymes, equipment and technology, enzymatic hydrolysis of lignocellulosic materials is safer and more precise than other methods. In essence, particular enzyme typically cellulose attack and destroy the cellulose in the fiber cell walls, resulting in the loss of the cell walls; this process produces single fine fibrils with significant hydrophobicity [38]. The most significant enzymes used to treat hemp fibers are lignin peroxidases, manganese peroxidases, and laccases, which function best at acidic pH levels. However, other enzymes, including Scourzyme L., function best at basic pH levels [79].

Fungi treatment: Lignin and hemicelluloses can be removed from sterilized hemp using the fungal treatment because the fungus enters the fibers' epidermis and release specialized enzymes at the insertion spots, resulting in fibers with enhanced hydrophobicity and noticeable roughness. To improve the adhesion between hemp fiber and polymer matrix, various fungi are used, including *Phanerochaete sordida*, *Pycnoporus species*, *Schizophyllum commune*, *Ophiostoma floccosum* (ascomycetes), and *Absidia* (zygomycetes) [79].

6.2 Matrix Modification Techniques

Since interfacial bonding between natural fibers and polymer matrices governs the mechanical properties of the fiber-reinforced polymer composites, researchers worldwide are working rigorously on improving the interfacial adhesion between the fiber and the matrix. Varieties of matrix modification methods are experimented to enhance the interaction between polymer matrix and fibers. Some of the remarkable techniques are discussed as follows [70]:

- Modification by coupling agents
- Modification via Epoxidation
- Modification by nano-reinforcement
- Modification by additives, plasticizers.

6.2.1 Modification by Coupling Agents

Generally, plant—based natural fibers such as hemp, flax, jute, and ramie are hydrophilic and polar due to their chemical structure. On the contrary the matrix is mainly hydrophobic and nonpolar. This incompatibility accompanies poor bonding between fibers and matrix. This fiber—matrix adhesion can be improved by introducing a coupling agent to modify the matrix which can act as bridging between polymer matrix and fibers, therefore, ensures the enhanced fiber—matrix interfacial adhesion as compared to the unmodified polymer matrix [17].

When making composites, coupling agents are added during and/or just prior to the impregnation process of reinforcement fibers with the polymer matrix. Generally, macromolecules with functional groups are used as coupling agents. These functionalized polymers' backbone needs to have compatibility with the matrix. Besides, these groups should be embedded to this backbone so that they can have interaction ability towards the reinforcements. Compatibility with the matrix is achievable by using identical or similar polymers as the matrix in the backbone of the coupling agents. On the contrary, interaction with natural fibers depends on non covalent/covalent bonds between the functional groups present on the natural fiber surface (such as -OH groups) and the coupling agents. The most significant coupling agents' group is embedded maleic anhydride polymer groups [102].

The most significant coupling agents' group is embedded maleic anhydride polymer groups. It has long polymer chains where maleic anhydride remains present as a functional group. For hemp or any other natural fiber reinforced polymer composites, polymer grafted by maleic anhydride is considered as one of the best coupling agent modified polymers. This grafted polymer works as a link between the polar fibers and the non-polar matrix. It creates chemical bonds with the fibers. Additionally, it creates a link with the matrix through the entanglement of the polymer chain [17].

6.2.2 Modification via Epoxidation

By converting the double bonds between the carbons into oxiranes [77], the matrix modification is also possibleWhile doing epoxidation, the different physical and chemical properties of the polymer change to such an extent in which percentage of mole alteration is launched. The modified matrix component becomes more polar with the increase of the degree of epoxidation. Hence, the compatibility of epoxied matrix with the natural fiber increases than the non-epoxied one. As a result, better bonding quality and better dispersion of filler between the epoxied matrix and fiber/fillers can be achieved [70].

6.2.3 Modification by Nano-reinforcement

Matrix modification is possible by adding nanoparticleswith a view to obtaining an increase in interfacial interaction. In this process, the matrix is customized by the integration of functionalized or virgin nanoparticles like zinc oxide nanoparticles, carbon nanotubes, titanium dioxide and nano calcium carbonate. Since these nanoparticles have amazing physical and mechanical features, they have been often used for modifying the matrix. In addition, by employing nanoparticles, chemical and physical anchor points are possible to achieve which results in excellent interfacial grip. Here, the synergistic impact has been achieved by following a strengthening mechanism. The full benefit of the nanoparticles can be grabbed, only if the dispersion within the matrix is homogeneous though achieving nano homogeneity is one of the major challenges in polymer nanocomposites.

Though the nanoparticles work as a tie between the matrix and the natural fiber thus, creating great adhesion, higher nanoparticles end up with agglomeration. This can affect the ultimate properties of the composite negatively. Hence, an optimal quantity of the nanoparticles is required to avoid nano agglomeration and structural voids as well as to ensure the enhanced interfacial adhesion that is responsible for composite properties [63].

6.2.4 Modification by Additives, Plasticizers

Additives, plasticizers are incorporated with polymers to obtain better polymer functionality with reinforcement fibers. Plasticizers are added to polymers to make them flexible, pliable, and processable. Plasticizers enhance the thermos-plasticity and fluidity of a polymer by reducing the viscosity of the polymer melt. The function of the Plasticizers is to break the connection between the molecules and to lessen the interface among protein chains. Plasticizers having polar groups tempts the hydrophobic features in the biopolymer matrixThe most utilized plasticizers are phytagel, poly vinyl alcohol, glutaraldehyde, and glycerol. The natural fiber composite with reinforced plasticized matrix shows less moisture absorption and the good interfacial shear strength thus an improved interfacial bonding In addition plasticizer modified polymers show less brittleness and improved flexibility properties [36].

7 Fatigue Behavior of HFRPs

7.1 Fatigue Behaviors of HFRPs

A lot of research have been conducted on the fatigue performance of natural fibers to be utilized as a replacement of glass fiber reinforced composites in various applications. Jute, flax, hemp, and sisal fiber are the most common to be used as reinforcement with thermoplastic and thermoset polymers in last few decades. HFRPs are the second highest used as structural components among all the NFRCs [98]. Although, the static tensile tests have revealed a slightly lower tensile strength and modulus of NFRCs than synthetic fiber reinforced composites, retention of higher tensile strength at higher cycles yields better performance of natural fibers in fatigue testing, especially in case of flax and hemp fibers [34].

7.2 Effect of the Fiber Characteristics on Fatigue Behavior

In case of natural fibers, the chemical composition and physical attributes of the fiber plays significant role on the static and fatigue tensile properties of the fiber reinforced composites. The strength of the NF highly depends on the cellulose content percentage of the fiber as well as the alignment of microfibril arrangement along the fiber, which allows the distribution of the tensile load more effectively. Having higher cellulose content and very low angle of microfibril with fiber direction provide the hemp fiber advantage over most of the other natural fibers reinforced composites by providing better tensile properties in static and fatigue loading [34]. Although the lower lignin content of hemp fiber attributes to higher moisture absorption, which may affect the fatigue properties over long periods.

7.3 Static Test

To study the fatigue behavior of natural fiber reinforced composites, studying the static axial loading properties is essential as the fibers collected from different location have divergent characteristics. The degradation of mechanical and physical properties of NFs at higher temperature limits their application as reinforcement of thermoplastics, thus most of the researchers choose to use thermoset resins to work with hemp fiber comparing to a few thermoset researches [98]. Table 2 depicts the tensile and compressive stress, modulus and strain to failure of various composites found by the researchers [61, 101]. Variation of resin material, fiber volume fraction and location of origin of hemp fiber yield different UTS/UCS and pertaining parameters for the composites. A linear increase in strength or modulus with respect to increased fiber volume up to 60% is reported by most of the researchers. Although, B. Venkatesh et. al reported a deterioration of tensile properties after only 10% of fiber volume fraction, it may be due to the use of mechanical stirrer for the mixing of fiber and resin [107]rather than conventional method of hand lay-up or compression molding. As moisture content plays a significant role on the HFRPs' behavior, testing at artificially created various environmental condition reveals the deterioration of tensile properties at higher moisture content and immersive conditions [16]. To improve the axial properties, fiber-matrix adhesion and sustainability various chemical treatment such as alkaline, successive acidic and alkaline as well as flame retardant treatment have been performed on HFRPs and tested at different conditional environment, which are summarized in Table 2.

7.4 Fatigue Test

As most of the structural components are frequently subjected to fatigue loading, improving the fatigue characteristics especially at higher cycles are the utmost priority. In HRPCs, improve of fatigue strength with increasing fiber percentage at a stress ratio of $R = 0.1$ in low-frequency fatigue testing, although no effect on fatigue sensitivity was observed. Moreover, reinforcement with hemp fiber also turns the failure mechanism during fatigue from ductile–brittle for control sample to full brittle for any percentage of fiber [35].

7.5 S–N Diagram

The semi-logarithmic graph of maximum strength over logarithmic value of number of cycle is primarily used for finding the allowable stress at any given cycle of loading. The fatigue sensitivity can be perceived from the slope of the S–N diagram. For various T-T stress ratio such as $R = 0.1, 0.3, 0.5$ the sensitivity varies lightly for hemp-polyester composite, whereas the C–C stress ratio of $R = 2.5$ shows less sensitivity than T-T ratios. However, highest sensitivity is depicted when T-C alternative stress ratio is applied of $R = -1$ [97]. Higher sensitivity to fatigue indicates higher degradation of the composites as well as lower ultimate strength at higher cycles. The lay-up angle of woven hemp fiber composites also shows variation in fatigue behavior of HFRPs. Despite of lower UTS, the ± 45° lay-up depicts lower degradation of tensile stress than 0°/90° lay-up demonstrated by higher slope value [29]. However, comparing to hemp woven fabric reinforced ones, UD hemp fiber reinforced composites portrays higher static and fatigue tensile stress as well as lower

Table 2 Static test results of hemp fiber reinforced composites

Resin	Fiber Volume fraction	Conditioning and treatment	Lay-up	UTS (Mpa)	UCS (MPa)	References	Source of fiber
Polyester	35.6%	–	$[0]_4$	171.3±6.5	95.1±6.9	[97]	Poland
Epoxy	4%	–	Used mechanical stirrer to mix	27.3	101.4	[107]	India
Epoxy	7%			34.2	107.6		
Epoxy	10%			46.2	115.2		
Epoxy	13%			30.4	91.58		
Polyester Scott-Bader Company	44%	–	Randomly oriented	53±6.0 6.2±0.6 1.39±0.26	–	[114]	UK
Polyester Scott-Bader Company	16.2%	–	Randomly oriented	20.1±3.6	–		UK
Polyester Scott-Bader Company	56.2%	–	Randomly oriented (CSM)	46.4±4.6	–	[100]	–
Epolam (Epoxy)	36±2%	–	[0°/90°]7	113±9	–	[29]	France
	36±2%		[±45]7	66±7			
PET	48%	–	0°	60±1.2	–	[57]	Italy
PE	–		0°	18±0.3			
PP	–		0°	28±0.5			
Unsaturated Polyester	56%	–	Randomly oriented (CSM)	46.4±4.6	–	[98]	–
	54%	10% at 24h		35.8±4.2			
	52%	10% at 1h		42±5.0			
	54%	10% at 24h +acetic acid		43.3±2.9			
	46%	5% at 24h		46.4±2.4			
	47%	1% at 24h		51.5±5.7			
Vinyl Ester	–	–	[0°/90°]5	60.897±0.93	–	[61]	–
		2688 h water immersed		48.341±1.76			
		Flame Retardeant treated		46.48±2.93			
		2688 h immersed+ flame retardant treated		35.191±1.63			
EPOLAM 2020	–	Ambient	[±45]7	60.0±3	–	[16]	France

(continued)

Table 2 (continued)

Resin	Fiber Volume fraction	Conditioning and treatment	Lay-up	UTS (Mpa)	UCS (MPa)	References	Source of fiber
		90 days in water		53.5±1.5	–		
		Dried after 90 days		53±0.5	–		

degradation of modulus over the fatigue cycles, because of significantly higher presence of fiber/resin debonding and cracking of matrix in woven composites [48]. Various model were used for fatigue life cycle prediction and those curve fittings revealed various values for fatigue sensitivity as shown in Table 3.

$$\text{Model no 1: } S_{\max} = \mathrm{S}_0\mathrm{N}^b \tag{1}$$

$$\text{Model no 2: } N_f = \left[1 + \left(\frac{\sigma_u}{\sigma_{\max}} - 1\right)\frac{f^\beta}{\alpha(1-R)^{1.6-\mathrm{R}|\sin\theta|}}\left(\frac{\sigma_u}{\sigma_{\max}}\right)^{0.6-\mathrm{R}|\sin\theta|}\right]^{\frac{1}{\beta}} \tag{2}$$

$$\text{Model no. 3: } \left(\frac{\sigma_0}{\sigma_{\max}} - 1\right)\frac{1}{(1-R)} = \alpha\left(N_f^\beta - 1\right) \tag{3}$$

$$\text{Model no 4: } \left(\frac{\sigma_\alpha}{\sigma_{ult}} - 1\right) = 1 - b\log N \tag{4}$$

Moisture absorption is one of the significant drawbacks of HFRPs, which can be attributed to the hydrophilic nature of natural fibers. To diminish the hydrophilic properties, various treatment can be considered like alkaline or silane treatment. In order to get a better picture of degradation, conditioning of the HRPCs by immersing it in water for 90 days and then testing it in high RH as well as drying for two days after immersion and then testing those in reference temperature was done. Wetting of HFRCs resulted in about 11% lower strength, yet 36% lower modulus value. Drying after wetting couldn't recover the strength much, but improved a little stiffness [16]. While 1% alkaline treatment flattens the S–N curve to ensure higher tensile properties at higher cycles, 5% alkaline treatment increases the UTS over the cycles while keeping the slope similar to control. Yet, 10% alkaline treatment provide lower properties than both 1 and 5% treatment [101]. Another limitation of NF is its flammability. After treatment with flame retardant, although the static tensile performance of the HFRPs diminishes as shown in Table 3, the fatigue sensitivity remains almost similar [61].

Table 3 Fatigue test results of hemp fiber reinforced composites

Resin	Condition	Frequency	Stress Ratio, R	Lay-up	Fatigue Life Model	Important Parameters		References
						S0	b	
Reichhold Norpol type 420–100 (polyester)	Ambient	10Hz	0.1[a]	$[0]_4$	Model no 1	196.4	−0.0623	[97]
			0.3[a]	$[0]_4$		234.8	−0.0548	
			0.5[a]	$[0]_4$		255.4	−0.0526	
			−1[b]	$[0]_4$		(161.7/ 50.5)	−0.1567/ −0.03	
			2.5[c]	$[0]_4$		(124.4)	−0.0373	
Vinyl Ester	Untreated	3Hz	0.1	$[0^\circ/90^\circ]_5$			−0.12	[61]
	Flame Retardant treated		0.1				−0.128	
						a	b	
EPOLAM (Epoxy)	Ambient	1 Hz	0.01	$[\pm 45^\circ]_7$	Model no 2	0.032	0.34	[16]
	Wet		0.01	$[\pm 45^\circ]_7$		0.029	0.3025	
	Wet/Dry		0.01	$[\pm 45^\circ]_7$		0.032	0.34	
EPOLAM (Epoxy)	Ambient	1Hz	0.01	$[\pm 45^\circ]_7$	Model no 3	0.106	0.169	[29]
			0.01	$[0^\circ/90^\circ]_7$		0.065	0.238	
Polyester	Hemp fiber only	1Hz	0.01	Randomly oriented	Model no 4	–	0.097	[100]
	Hemp skin-glass core hybrid						0.115	
	Glass skin-hemp core hybrid						0.115	
Polyester	1% alkaline treated	1Hz	0.01	Randomly oriented			0.070	[98]
	5% alkaline treated						0.063	
	10% alkaline treated						0.087	

During the lifetime as structural components, the HFRPs may experience sudden low or high velocity impact. That's why the fatigue performance after impact has been tested and reported. Surprisingly, the impacted HFRPs display flatter S–N curve than the control one despite of having much lower residual stress to start as depicted in Fig. 20 [114]. In order to improve the fatigue performance, hybrid glass and hemp

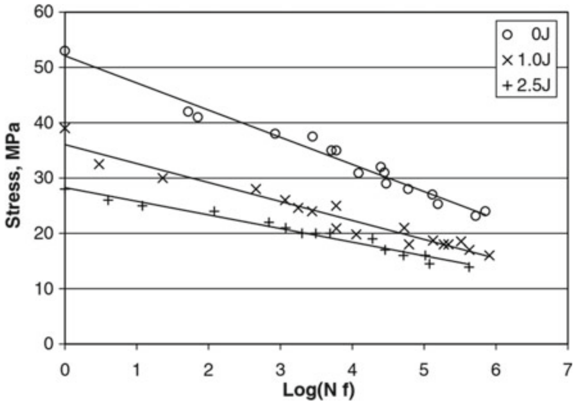

Fig. 20 S–N diagram of hemp fiber reinforced polyester composites; undamaged and after impact of 1J and 2.5J; reused with permission from [114]

fiber reinforced composites are fabricated. These hybrid composites shows steeper S–N curves than control, yet the UTS over the lifecycle yields higher value [100].

7.6 Hysteresis Loop Analysis

The hysteresis loop analysis tells a lot about the degradation of the composite over each cycle. Like most of the NFRPs, the dynamic modulus and minimum strain of the HFRC follows a common trend of higher minimum cycle strain increasing rate and rate of loss of modulus at the first 10 to 15% of cycles, almost constant rate in the middle section comprising of 70 to 80% of the total life cycles and final 10 to 15% of the cycles with rapid rate of strain and modulus change exactly before the final failure. While 0°/90° lay-up shows the typical curves, the ±45° lay-up depicts much higher rate of change in the middle section also revealing major damage even in the middle cycles [29]. Experiment at conditional environment, gives another perspective of analysis. The maximum stress of fatigue testing plays a major role while experimenting at wet and dried after wetting condition. At higher fatigue stress, loss of dynamic modulus of wet condition is slightly less than the reference condition ones in the first few cycles, whereas a lower fatigue stress loss of dynamic modulus of wet conditions are severely higher than the reference conditioned ones, demonstrating major degradation in wet condition in case of lower fatigue stress as shown in Fig. 21 [16].

7.7 Constant Life Diagram

Constant life diagram is generally produced by extrapolating the fatigue life-cycle data at different stress ratio establishing relationships among life cycles, mean stress

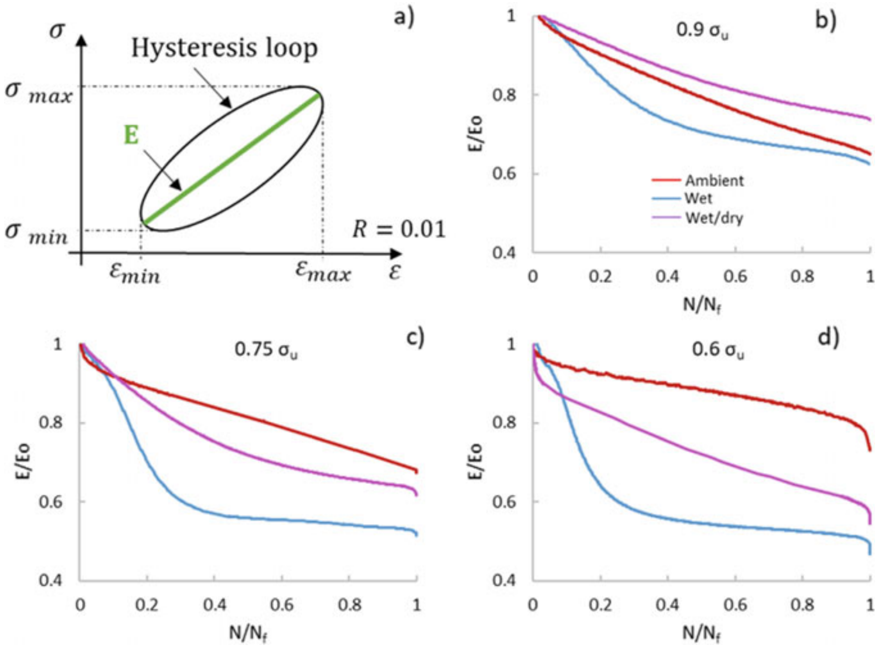

Fig. 21 **a** Schematic representation for calculating dynamic modulus. Depiction of normalized dynamic modulus vs the normalized cycles number for stress level of **b** 0.9, **c** 0.75, **d** 0.6; reused with permission from [16]

level and amplitude of stress. Various stress ratio of R = 0.1, 0.3, 0.5, T-C ratio R = −1 and C–C ratio of R = 2.5 have been taken into account to form a constant-life diagram for hemp fiber reinforced polyester composites as shown in Fig. 22. From the diagram, it is evident that, the most crucial loading condition for hemp fiber is tensile-compressive. Although the static compressive strength yielded really lower value, fatigue performance of compressive-compressive loading condition depicts comparable performance of tensile-tensile loading conditions. The fatigue sensitivity of NFRPs generally depends on the resin type rather than the fiber properties, so the generated constant-life diagram can be applied approximately to most of the NFRPs [97].

8 Challenges in HFRPs

Though HFRP has a notable assertive impact, some challenges will also be faced during the composite material preparation procedure. As hemp is a natural fiber, it has some problems related to the fiber structure and properties in case of incorporating it in composite materials [98]. Lack of adequate uniformity and smoothness in

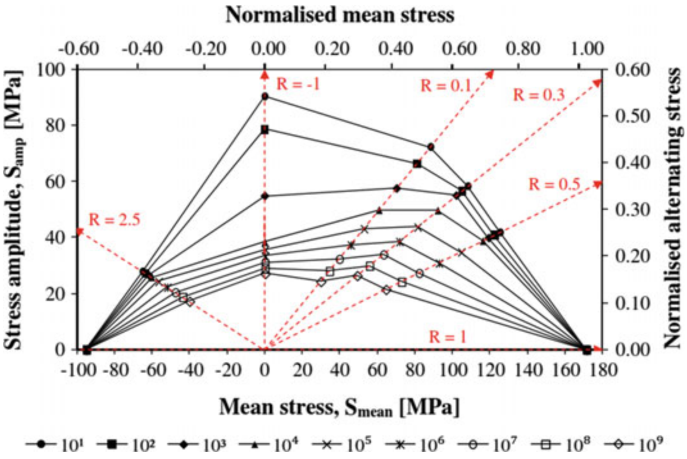

Fig. 22 Constant-life diagram for Hemp reinforced polyester composites; reused with permission from [97]

fiber surface is considered a significant drawback in HFRP composites. The morphological irregularity of the hemp fiber surface is a problem for improving the interfacial adhesion between the fiber and polymer matrix while preparing a composite material. According to the experimentation of Mutje et al., the polarization nature of hemp fiber was found significantly strong compared to other possible synthetic alternatives like glass fiber, polypropylene (PP) etc. [66]. To ensure good compatibility with the non-polar polymer matrix, which is hydrophobic in nature, polarized cellulosic fibers like hemp requires impactful surface modification. Though surface modification ensures a fruitful stress transfer process from the polymer matrix to reinforced fibers in composite materials, the process usually causes degradation in the material's toughness [93].

The brittleness of the HFRP composite was also incremented in some cases, which significantly hampers the effectiveness of the composite material. Sebe et al. showed this type of surface modification by accomplishing the esterification of hydroxyl groups presenting the hemp fibers in the presence of polyester as a matrix material. As a result, they experienced the mentioned type of brittleness in the composite [93].

The tensile modulus and strength of hemp-reinforced composites with PP were lower than glass-PP based composite materials. Diversified compatibility agents like the maleated polypropylene (MAPP) application can be a solution to deficient tensile properties of hemp-PP based composites [66]. Like most other natural fibers, the impact strength of HFRP composites is remarkably lower than glass fiber-reinforced polymer composites. The insignificant properties of flexural creep resist HFRP from being the considerable choice of material under the high fatigue load condition of polymer composites [89]. Variability in mechanical properties also impacts the durability of hemp fiber, constraining the HFRP from becoming the most suitable one for long-term purposes [87].

Accelerated moisture absorbency can lead a fiber towards unwanted swelling and microbial attack. With a moisture content of 5%–10%, the fiber acts highly hydrophilic, this results in an impactful change in the dimension of the fibers along with the variation of significant mechanical properties. As a result, the process ability of the fiber becomes disrupted and porous. The impact of swelling of fiber molecules in the interfacial bonding between fiber and matrix is provided in below mentioned Fig. 23 [8].

Among novolac resin-reinforced banana, sisal and hemp fibers, Mishra et al. found the maximum moisture and steam absorption property in hemp fiber [59] This parameter significantly reduces the antimicrobial property of the HFRP composite and makes it highly sensitive to microorganisms. Due to this significantly higher moisture content, hemp fiber reinforcement tends to crack in the composite materials.

HFRP composites also face some challenges from thermo-mechanical consideration. The low thermal stability of hemp is a considerable obstacle in HFRP [56]. Tajvidi et al. found that the Tg (glass transition temperature) became lower in composite materials, making the reinforcement procedure challenging [106]. The property of thermal instability made hemp fiber less considerable for thermo-mechanical composites. Hemp fiber initiates thermal degradation and significant weight loss around the temperature of 200 °C, which become more rapid with

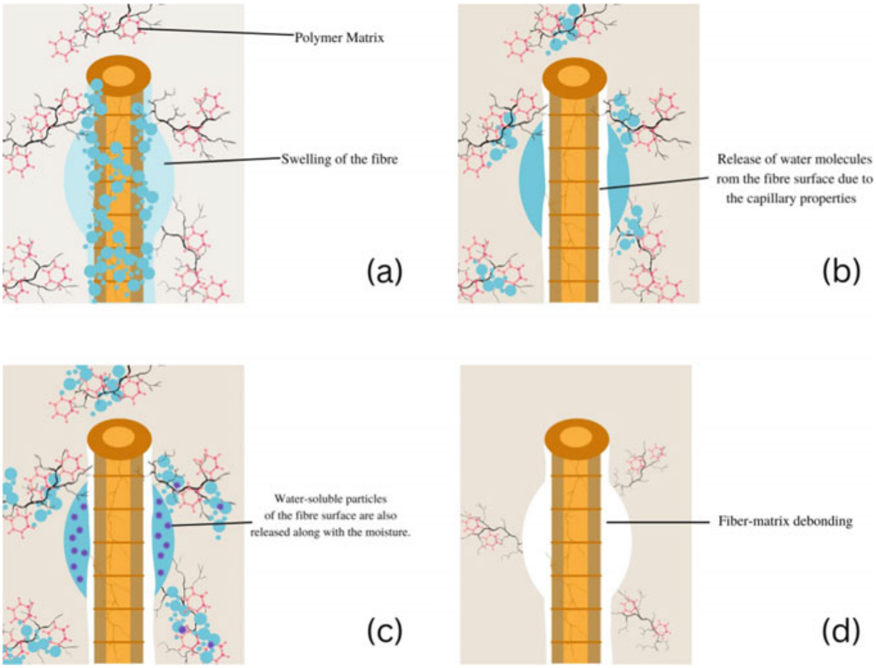

Fig. 23 The mechanism of interfacial bonding creation between fiber and matrix: **a** Swelling of the fiber due to the moisture absorbtion of hem-cellulose, **b** Release of the moisture from fiber surface, **c** discharge of the water molecules and debonding occurs, **d** Deponding of the fiber and matrix

increasing temperature. At high temperature, hemp fiber lost its moisture and face notable reduction in fabric weight [17]. The incremented amount of hemp fiber reinforcement in composites made them more insignificant considering the thermal properties.

Natural fibers like hemp have excellence in various dimensions as a reinforcement material in polymer composites. However, there have some challenges as well. Poor adhesion and interfacial bonding restrict hemp fiber from becoming the best choice as reinforcing fiber. The handling and control of the properties of HFRP are complicated due to the irregular surface morphology. Higher moisture absorption can be liable for swelled, porous, microorganism-prone composite materials. Insignificant thermal instability restricts it to be the choice where dealing with heat, and thermal energy is the main issue. As a result, HFRP is not considered the best choice for long-term usable composite preparation. Their low aging properties are responsible for making them unsuitable in diversified sectors.

Though HFRP has some challenges like all other natural fiber-reinforced composite materials, these challenges can be solved or reduced in many cases by performing some physical and chemical treatment. In addition, selecting an accurate processing technique by considering the desired result in the final material can resolve the challenges significantly and help to get the best possible outcome in a sustainable manner.

9 Conclusion and Future Perspective

Due to advantageous mechanical properties, affordable cost, assertive impact on the environment, biodegradable nature, and notable durability, hemp fibres are a potential fibre for reinforcement in polymer composite nowadays. Though hemp fibre has significant advantageous parameters, some constraints can also be found when considering hemp as a substitute for synthetic-based high-performance fibres. Factors like hydrophilic nature tend to create complications to ensure adequate interfacial bonding between the reinforced fibre and polymer matrix. By implementing diversified techniques and modifications, the scenario can be improved to a satisfactory level. A suitable fabrication technique and an adequate manner of fibre surface or polymer matrix modification are necessary to achieve an effective outcome. In this chapter, the fabrication techniques of hemp were discussed thoroughly along with the elementary conceptualization regarding the hemp fibre and polymer matrices.

Consequently, hemp fibre and matrix modification techniques are discussed to improvise the interfacial bonding capability between the fibre and the matrix. The impact of interfacial bonding on the polymer composite's fatigue behavior is also briefly discussed. The challenges hemp fibre can face in the polymer composite industry were also mentioned. Difficulty in adequate interfacial bonding due to the hydrophilic nature of the fiber is a significant matter which preparing a composite. However, the hemp fibres can be used after necessary modifications to achieve a positive outcome in an environment-friendly manner. Diversified modern and effective modification technique adaption can be the future direction that can facilitate the useability of hemp in the composite industry.

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Effect of Interfacial Bonding Characteristics on Physical, Mechanical and Fire Performance of Bamboo Fibre Reinforced Composites

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Abstract This chapter reviewed the influence of interfacial bonding characteristics of bamboo fiber on the composites' mechanical, physical, thermal, and fire performance. Because of their complete biodegradability and renewability, being economical, non-toxic, non-abrasive and environmentally friendly, having high aspect ratio, socio-economical advantage and strong mechanical performances, bamboo fibers are considered promising reinforcements for polymer composites. As an alternative to petroleum-based or synthetic materials, bamboo also has the potential to be used in biopolymer composites, which can be utilized as construction, building, architectural and other advanced materials. However, strong and substantial interfacial bonding of the bamboo fibers with the polymer matrix is required to create high-end load bearing composites. Therefore, it is critical to achieve a good fiber-matrix interaction, resulting in lesser voids and better adhesion and mechanical properties. Thus, most bamboo fibers are treated, hybridized, laminated, and coupled using chemical agents to enhance the fiber-matrix bonding, which in turn enhances the performance. The detailed understanding of the aspects of fiber-matrix interaction and their respective influences on the various properties of the bamboo composites are given in this chapter, along with the superiority of the bamboo fibers in being used as the polymer reinforcement.

Keywords Interfacial bonding · Bamboo fiber reinforced composites · Physical performance · Mechanical performance · Thermal performance · Fire performance · Bamboo · Composite

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1 Introduction of Bamboo

Among perennial grasses, bamboo is one of the fastest growing and strongest ones. However, it should be noted that bamboo grows much slower than grass. Bamboo is widespread across the globe and is a member of the family *Poaceae* and subfamily *Bambusoideae* [1, 2]. More than 120 genera and around 1700 species of bamboo are mostly found in North, Central, and South America, Africa, and Asia [3]. Since antiquity, bamboo has been widely used in South America and several Asian countries as a sustainable substitute for wood, being frequently used even in buildings. Table 1 shows the coverage of bamboo in the world. Wood can take up to 20 years to grow, whereas bamboo grows to maturity in only about three years, with tensile strengths comparable to mild steel. The fastest-growing bamboo species grow vertically about two inches in an hour, while there are some specific species (Moso bamboo) that can reach heights of 60 feet within just three months [4]. Bamboo has an astonishing rate of development, causing no harm to the ecological equilibrium due to extensive removal and cultivation. Bamboo products have seen a steady and significant increase in demand for various applications. Scarcity of wood in many countries across the world has helped in this increased demand for bamboo, which is proven to be a great alternative to wood, especially due to its growth rate and mechanical properties (Fig. 1).

Generally, the use of bamboo as building materials, ornamental accents, and panels, as well as its production, distribution, and commerce, negatively influence the environment and the global economy. Although bamboo is technically grass, it is sometimes referred to as the "poor man's lumber" [7]. The Forest Resource Assessment by FAO in 2015 recognized bamboo as being an intricate part of the forest [8]. Bamboo may also be utilized successfully for carbon sequestration. Therefore, their products may be the best substitute for the growing concern about global warming and the ongoing destruction of tropical forests [9]. Planting bamboo could help faster transfer to a circular economy by offering a practical, bio-based substitute for materials that are not renewable and have a high carbon footprint [10]. Thus, bamboo is often considered a tree or plant for productive forests.

Planting productive forests with foreign tree varieties is an engrained venture in southwest Europe and the Mediterranean [11]. Plantations offer a chance to diversify

Table 1 Countries and regions with bamboo coverage [5]

Countries	Bamboo regions	Percentage
Thailand, Sri Lanka, Philippines, Malaysia, Korea, Japan, Indonesia, India, China, Cambodia, Burma, Bangladesh	Asia–Pacific Region	65
Some European Countries, Venezuela, Nicaragua, Mexico, Honduras, Guatemala, Columbia, Costa Rica, Brazil	America Region (Latin America, South America, and North America), European Region	28
Eastern Sudan and Mozambique	African Region	7

Fig. 1 Bamboo (*Gigantochloa scortechinii*) strips from Malaysia [6]

financial portfolios even though they require a sizable investment, a lot of labor, and substantial capital to establish productive plantations [11]. From this angle, bamboo farming has been more popular and has a growing demand recently in Europe. Species like *P. pubescens*, used for landscaping and producing wood, fibers, shoots, and biochemical components, among other things, are particularly prized for their various uses [12]. In addition, Europe has also benefited economically and environmentally from growing bamboo. Bamboo plantations have a few special characteristics, such as the fact that they need less upkeep and are more ecologically friendly than other crops since they don't use pesticides. Furthermore, while most wood species require 10 to 50 years to attain maturity in the climate of Europe, bamboo plants need just 5–7 years [13].

Additionally, maintaining bamboo requires little work and less money, with the primary requirements being weeding during the first two years, adding organic fertilizer, and watering. Bamboo offers a potential financial investment in Europe's energy crop. Bamboo may flourish on damaged and marginal soils because of its extensive rhizome and fibrous root systems, dense leaves, and green mulch, stabilizing the soil, reducing erosion, and retaining water [14]. Contradictions are common in studies on bamboo's potential for the environment. Although some researchers assert that one of the critical reasons behind growing cultivation of bamboo across the world is due to it being a more environmentally friendly substitute to current forestry practices, it is important to evaluate its eco-friendly impacts when cultivated for profitable reasons [15]. Bamboo spreads primarily via the growth of rhizomes. As a result, the location of the plantings must be considered in the neighborhood. For instance, preventing bamboo from expanding into ecologically sensitive areas would be important. In addition, production sites need long-term management measures to lessen the number of abandoned bamboo plots that might be a source of invasive bamboo species [16].

China, Africa, India, South and Central America, and other places where bamboo is a native plant have received most of the attention in economic studies of bamboo plantations [5]. Bamboo's economic advantages are frequently considered as are its ecological advantages, thus connecting both the defensive and offensive roles [17, 18]. Examples include the "twofold potential" of using bamboo, (1) creating wood and shoots, and (2) storing carbon, resulting to carbon credits. The latter feature might also improve farmers' financial situation [19–21]. However, according to [22] and [23], bamboo is more sustainable compared to conventional crops. Its Net Economic Benefit (NEB) is lower than the common crops, especially in the initial years after its release. A bamboo intercropping system is the greatest tactic for small-scale farmers in Ghana, according to [24], because it can boost system efficiency, diversify sources of income, and uphold environmental sustainability.

2 Economic and Socio-economic Aspects of Bamboo

Studies on investments in bamboo plantations in Europe are few, and a similar goes for the United States. Most researchers concentrated on the advantages and disadvantages of growing bamboo in Western Europe, especially the economic viability of using bamboo to produce biomass and the potential adaptation of bamboo as a Western European building material [25]. Even though North America, France, Italy, and other European countries have not studied the economics of bamboo, the engineering and technical elements of products created from Italian plants have been recently investigated for the past few years [9, 26, 27]. Many factors have led to bamboo's rising popularity in Italy and other places in Europe, focusing on the southern part where certain bamboo species flourish in the Mediterranean climate [28]. Two case studies from Calabria look at the viability of bamboo from a commercial and eco-friendly standpoint. Based on bibliographical research and in-person interviews, a SWOT analysis was conducted on the variables influencing farmers to plant bamboo in southern Italy.

Europe purchases 37% of bamboo goods sold globally, making it the biggest importer of bamboo products worldwide. On the other hand, Western Europe has a mixed perception of bamboo. It provides ecological knowledge and concern for the environment, on the one hand, given its natural origin and the little environmental impact of the development process. On the other hand, it is often regarded as a material of poor value [29] due to problems with numerous areas of its development (marketing, management, organization, and product development). There ae several steps in the manufacturing chain of bamboo goods (harvesting, processing, marketing, transport, plantation establishment, and management) resulting in a small market share in the west. From 2015 to 2019, the Netherlands has been the top importer with 72,020,545 €, proceeded by Spain with around 40,250,401 €. Italy is third on the list with 34,756,846 €, followed by the United Kingdom with 24,771,569 €.

France was the biggest importer of bamboo flooring between 2015 and 2019, values at 22,751,771 €. Then comes United Kingdom at 21,452,636 €, followed by Germany at 14,074,743 €, and Belgium at 10,804,083 €. The Netherlands was the top importer of bamboo panels (HS code 44,120) over the same time. Germany again comes second with a valuation of 11,524,530 €, followed by Belgium at 9,660,412 €, and France at 8,766,913 €. The EU Timber regulations (EUTR) cover all imported goods. Therefore, it is obvious that initiatives to grow bamboo in Europe has increased to satisfy this demand and reduce the environmental costs associated with shipping the product from its source countries. [29].

Every continent has a native bamboo population, except for Europe and North America, where cultivation is still in its experimental stages. However, statistics on bamboo are scarce compared to most other commodity crops since the domestic bamboo markets are still largely unorganized. In addition to other species like *P. edulis* and *P. viridiglaucescens*, certain pilot plots of *P. aureosulcata 'Spectabilis,' Phyllostachys vivax*, and *P. violascens* (syn. *P. praecox*) is situated in Belgium and Germany. These two last species are also cultivated in France with new *P. edulis* plants are emerging in Ain and Romania with around 30 hectares.

Ireland is home to plantations of *P. humilis*, *P. bissetii*, *P. mannii* (syn. P. *decora*), and *P. aurea* [30]. Even while most cultivated bamboo and its processed products are sold inside Europe, interestingly, although being a sizable importer of raw bamboo, European countries are also the world's second-largest exporter of bamboo goods, especially to Germany and France. Over 40% of the entire value of European exports went to these two nations, whereas just 8% went to China and Angola, where most of the exports were processed goods. Italy has drastically increased their bamboo import over the last three years, especially in the form of raw materials and furniture. It implies a demand for bamboo and the potential to establish a regional supply chain. The sales of veneered wood, plywood, and similar laminated bamboo wood increased significantly between 2016 and 2020, from 56,513 € to 135,848 €, as shown by ISTAT statistics [31]. Since 2014, several manufacturing consortia and cooperatives have been established, expanding the bamboo market that bears the "Made in Italy" or "Made in Europe" badge. On the other hand, there are numerous records of much older bamboo plantations, such as the 22 hectares of the bamboo plantation at Selva di Paliano in Frosinone (Rome, Lazio), the 1-hectare bamboo plantation in Lucca (Tuscany) [32], and the 0.5-hectare bamboo plantation in Syracuse (Sicily) [33]. Lucca (Tuscany bamboo plantations are currently around 70 years old, while Syracuse (Sicily bamboo plantations are around 60 years old [32, 33].

Currently, across Italy, the CBI (Consorzio Bambù Italia) has planted roughly 2,000 acres of bamboo [34]. According to [28], the primary species produced by the CBI are *Dendrocalamus brandisii,* a sympodial species that are suited to the tropical climates having pronounced dry seasons, and *Phyllostachys edulis* (Moso bamboo), part of the monopodial species that live naturally in temperate regions with cold and wet winters. The Moso bamboo is grown mostly for its shoots and a total of

148 hectares have been planted as part of plantation runs by various organizations in Italy, including Calabria (59 ha), Emilia Romagna (3 ha), Basilicata (35 ha), Marche (1 ha), Piedmont (35 ha), Friuli Venezia Giulia (2 ha), Lombardy (10 ha) and Tuscany (3 ha). They may also be found in Lake Maggiore, Lake Como, and Lake Garda in northern Italy. Between Cuneo and Trieste, there is a region with a moderate climate and comparatively heavy summer rainfall. The growing of bamboo, including species like *P. bambusoides*, *P. iridescens*, and *P. vivax*is, is extremely common in the Piedmont area. Molari et al. [27] tested the mechanical characteristics of the culms of these species. Various bamboo species have been seen becoming invasive and naturalizing in this area. There are many bamboo groves, notably those of *Phyllostachys nigra* and *P. aurea*, all over central Italy until Naples, particularly in Frosinone. Lazio, Lombardy, and Tuscany have *P. bambusoides* plantations of about 30 hectares. *P. viridiglaucescens* can further be seen in some places of Tuscany, which is an intriguing species because of its morphological traits [26]. In the United States, most of the bamboo planted is from the species of *P. nigra 'Henon.'*, *P. rubromarginata, Bambusa Balcooa, and Dendrocalamus asper.* Most of these species are grown in Florida and California, United States Bambu [35]. Bamboo farming is still in its early stages in many non-origin places throughout the world, i.e., Italy, France, United States, and there is a lack of trustworthy information on the traits and yield of the crops [36]. The demand for raw materials and related goods is not estimated either.

Despite this, a nationwide manufacturing chain is being built with several important players, including tissue culture and nursery operations, industrial growers, and specialty merchants. Although the export of raw bamboo materials decreased, this decline in export may be related to domestic bamboo use [37]. China is the top exporter to the US and the EU. The main beneficiaries of cultivating and harvesting bamboo are the farmers. The consistent source of income from growing and harvesting the bamboo has improved their ability to handle pressure if there is a loss in bamboo sales and their basic cultivation skills. An unproductive piece of ground may be turned into productive land thanks to the ecological system, which also reduces landslides and soil erosion. Bamboo growing techniques help impoverished rural farmers improve their standard of living. By interplanting bamboo with other food plants, it is possible to improve food security while simultaneously protecting the environment and damaged land. The cheapest way to cultivate bamboo is by cultivation, the most expensive ways include bamboo propagation, land, and labor. Undoubtedly, the socio-economic advantages of producing durable consumer goods via the creation of products like furniture, flooring, bamboo-based composites, fences, and other decorative items significantly impact the economic growth of many nations. Natural resource depletion and rapidly rising crude oil costs have sparked interest in using bamboo in composite technologies.

Strict laws requiring the design of eco-friendly consumer goods are pressuring industries to develop a method for using renewable resources, one such intriguing one being bamboo. Bamboo is one of the best resources that can be utilized as a composite reinforcement in place of glass fibers, which is sourced from depleting natural resources [38]. Researchers are searching for a more environmentally friendly way to address this environmental hazard. A new age in the composites sector has been sparked by simple access to bamboo. To avoid using wood, technical, and regulatory attempts to employ bamboo composites in the public interest are also moving quickly. Developing composites made from bamboo for household goods, transportation, and construction has given the bamboo economy a new direction while helping the average person economically and socially. In addition, promoting bamboo-based composites has opened up new job opportunities, and policies are being implemented all over the globe to increase public interest by suggesting various measures, such as exempting bamboo composites from excise taxes [39].

3 Fiber Matrix Interaction in Composites

An important role is played by the interfacial bonding between the fiber and matrix, in any composite, due to the stress being transferred through them. Thus, a good bonding at the interface is necessary to achieve ideal reinforcement and control the composites' mechanical properties. Full contact that promotes fracture propagation might also diminish strength and toughness [40]. Interfacial bonding is generally poor if hydrophilic fibers and matrices are combined, resulting in hydrophobicity and lowering the mechanical properties with unfavorable long-term effects. Due to the interfacial connection between fiber and matrix, wettability is a key prerequisite to their bonding. Fiber wetting would be a stress concentrator and might result in interfacial damage, impacting the composites' flexural and tensile strengths. Nevertheless, physical and chemical treatment may improve interfacial strength and fiber wettability. Interfacial bonding includes mechanical interlocking, electrostatic, chemical, and inter-diffusion bonding. The rough fiber surface and strong interfacial shear strength cause mechanical interlocking, which does not affect the transverse tensile strength. However, only the metallic exterior will experience electrostatic bonding [41].

In general, raw bamboo fiber and epoxy polymer have insufficient interfacial adhesion, which lowers the strength of the composite. One way of improving this is by removing the extra moisture through chemical and heat treatments, which can strengthen the interfacial bonds between the two interphases, improving the mechanical interlocking. The bonds get strengthened when reciprocating chemical groups are present in the fiber surface and matrix, causing chemical reaction and helping enhanced bonding. Therefore, it often makes sense to use a coupling agent that links the fiber and matrix to enable that chemical bonding. It is further explained by the inter-diffusion bonding between the fiber and matrix due to the interaction of the surface atoms and molecules. According to the findings of [42], chemically treating bamboo fibers for about an hour with 4 wt.% alkaline solution has detrimental effects on the fibers' tensile strength. In addition, the chemical treatment removes lignin and hemicellulose and improves the surface area's efficiency for a strong connection with the matrix. However, it is also said that a greater NaOH concentration can deteriorate the bamboo fiber characteristics if it is not properly controlled.

Chemically altering fiber is necessary to increase the amorphous nature of cellulose at the cost of its crystallinity. Hydrogen bond, available in the network structure, gets eliminated, which forms another significant anticipated change during the process. Mercerization, which is another name for alkaline treatment, is one of the many available chemical procedures that is generally utilized for strengthening both thermosets and thermoplastic composites. The fiber bundle is divided into smaller pieces during mercerization, a process known as fibrillation. As a result, reduced fiber diameter and increased aspect ratio create rough surface topography, improve adhesion at the fiber-matrix interface, and enhance mechanical characteristics [43]. In addition, the alkaline oxide will be produced due to the outer hydroxyl groups, which are generally unstable, that disintegrates and reacts with water to destroy the reactive ionized molecules. Consequently, it is anticipated that cellulosic fiber's surface roughness will be improved, and hydrophilic groups will be eliminated [44], resulting in a chemical reaction as shown in Eq. (1):

$$Bamboofiber - OH + NaOH \rightarrow Fiber - O - Na^{+} + H_2O \tag{1}$$

Many known chemical modifications have been applied to not only bamboo but also to other lignocellulosic fibers to increase the fiber matrix interaction. Some critical ones that are important and can potentially be applied to bamboo, a lignocellulosic material, are detailed in this section. Silane was applied to kenaf fibers to increase cross-linking both at the interface and the fiber surface regions, strengthening the fiber-matrix bonding [45]. Furthermore, the bi-functional groups present in the silane molecule may interact with the fiber and matrix, forming a siloxane bridge, resulting in an enhanced and powerful contact between the two while improving the mechanical performance of composite materials. In moist conditions, hydrolyzable alkoxy groups form silanols, reacting with the fibers' hydroxyl groups, as illustrated in Eq. (2) according to [46]. During the procedure, the fiber surface adsorbs the silanols chemically, creating stable covalent bonds in the cell walls. The Eq. (3) demonstrates how adding silane results in the formation of hydrocarbon chains, limiting fiber expansion. The covalent contact between the matrix and the fiber enables the cross-linked network. As for the chemical reaction, it can be represented as:

$$CH_2CHSi(OC_2H_5)_3 \rightarrow H_2O\,CH_2\,CHSi(OH)_3 + 3C_2H_5OH \tag{2}$$

$$\begin{aligned} &CH_2CHSi(OH)_3 + bamboo\,fiber - OH \rightarrow CH_2CHSi(OH)_2O \\ &- bamboo\,fiber + H_2O \end{aligned} \tag{3}$$

Using inorganic fillers in polymer matrices often results in unstable dispersion with compatibility issues, which can be limited through surface treatment of the fibers and polymer. Successful grafting of the organic monolayers onto the fibers reduces the likelihood of accumulations and enhances the dispersity simply by modifying the hydrophilic qualities to the hydrophobic ones [47]. On the other hand, the presence of silane in the fiber-matrix interphase is often considered to act as an agent or catalyst that helps in bridging or bonding, promoting adhesion between two incompatible materials. The influence on silane can be attributed to the complex chemical and physical interactions that directly affect the strength, perseverance and adhesion between the components of the composite. Therefore, coupling agents that are made from silane, with unique physical and chemical aspects will help strengthen the bonds at the interphase [48]. Heat therapy, corona, plasma ultraviolet (UV) and electron radiation are some additional forms of physical therapy.

Corona treatment is accompanied by high voltage applied to the tips of sharp electrodes that are separated at low temperatures with the help of quartz, whereas air pressure produces the plasma, which together helps increasing the surface roughness and polarity. On the other hand, plasma ultraviolet therapy is usually conducted inside a vacuum chamber, as it requires accurate composition of pressure and gas, successfully maintained with the help of a constant supply of gas. The fiber surface gains hydrophobicity through this process and increases surface roughness, including interfacial adhesion. An earlier study established that the plasma UV treatment in beneficial in effectively increasing the interlaminar shear and flexural strengths of the composites by 35 and 30%, respectively. Heat treatment, which involves heating the fiber to the fiber degradation temperature, can also change the fiber's chemical, mechanical, and physical properties [49].

4 Bamboo as Reinforcement in Composites

The research and production of polymer composites must now utilize natural, recyclable materials because of the rising costs of synthetic and common polymer raw materials, natural reservoirs' long-term viability, and environmental dangers [50, 51]. In the past, synthetic fibers dominated the reinforcement market, however, natural fiber reinforcement has acquired significant momentum as substitutes for artificial fiber in various applications. Using natural fibers with polymer matrices made from renewable and non-renewable (petroleum-based) resources have gained further popularity over the past ten years in an effort to compete with the widely produced synthetic counterparts [52].

To compete and gain market share in the current market that is primarily dominated by synthetic products made of petroleum or petroleum-based aspects, it is critical for the renewable and biodegradable polymer composites to have similar or enhanced performance [53]. Researchers have used softwoods and hardwoods to harvest the necessary fibers to reinforce different composites. Natural fibers have a significant economic role for several developing nations. Examples include sisal in Tanzania, jute in Bangladesh, and various West African nations' cotton [50]. Research and development on polymer composites have been conducted in nations with scarce agricultural and forestry resources. One of the agricultural products that may be used in creating polymer composites is bamboo [54]. Asia and South America are both home to a large bamboo population. Although bamboo is a naturally engineered material, several Asian nations have not fully exploited its potential. Since it takes only months for this sustainable substance to develop fully, it has become the foundation for many societies' socio-economic standing. Due to its excellent strength-to-weight ratios, bamboo has historically been used in various homes and equipment. This property results from the longitudinal orientation of the fibers. Therefore, it is possible to build composites reinforced with bamboo fibers [55, 56]. Although bamboo fibers have superior mechanical qualities naturally, they are more brittle than other natural fibers because of the additional lignin that covers them.

The applications and possible use of bamboo as the reinforcing fiber in polyer composites are immense. This can be attributed to its structural variety, mechanical attributes, fiber extraction, chemical modification, and thermal properties [57]. According to [58], bamboo has a comparatively small microfibrillar angle of 2°–10° and contains a high concentration of lignin and 60% cellulose. This distinctive quality has led to the use of bamboo fiber as reinforcement in several matrices [58]. Researchers have created several techniques to harvest bamboo fiber for composite reinforcement. Alkaline treatment can facilitate extraction and optimize bamboo fiber separation to prepare bamboo fiber-reinforced polymer composites [59, 60]. Researchers investigated the changes in bamboo fiber's fine structure brought on by exposure to various alkali solution concentrations [61]. Researchers also examined the impact of bamboo fiber mercerization on bamboo composites' mechanical and dynamic mechanical characteristics in an intriguing study [61, 62]. The primary aim is to have bamboo reinforced polymer composites that have similar or superior qualities compared to that of synthetic fibers. Twin-screw extruders have often been utilized to combine and process biodegradable polymers and bamboo fibers as reinforcements to fabricate bamboo-reinforced polymer composites. Researchers have also used orthogonal bamboo fiber strip mats to create composites with epoxy and polyester using the hand lay-up method [63, 64]. Short bamboo fiber-reinforced epoxy composites can be made using dried bamboo fibers, and the relationship between the fibers' tensile and chemical resistance qualities has been reported [65]. To produce polypropylene composites reinforced with bamboo fiber, researchers employed *Bambusa Paravariabilis*, a bamboo type widely cultivated throughout Asia [66]. Another fascinating study combined E-glass and bamboo fibers, commonly grown in Southeast Asia and widespread in Singapore, as reinforcement in hybrid composites [67]. Researchers looked at how fiber length affected the mechanical properties of polymer composites using starch resin and short bamboo fibers [68].

Using a steam explosion procedure to separate the bamboo fibers from raw bamboo trees, bamboo fiber-reinforced polymer composites' mechanical properties have also been evaluated [55]. Green composites that are biodegradable and environmentally benign have been created using bamboo fibers that are micro- or nano-sized and have a reasonable amount of strength and stiffness [69]. Bamboo and biodegradable resin are combined to create bio-based polymer composites that have also been examined for their flexural characteristics and compared with composites manufactured from kenaf [70]. The authors then calculated the flexural modulus using Cox's model, which considers the effect of fiber compression. The outcomes and experimental data were in good agreement.

The morphological and mechanical properties of the bamboo flour-filled HDPE-based composites were also examined. It is focused on the crystalline nature of the malleated elastomer modifier, combined EPR-g-MA and PE-g-MA modifier systems, and the loading rate of bamboo flour in the presence of the combined modifier [71]. In addition, researchers examined the thermal characteristics of jute/bagasse hybrid composites and found that increasing the char residue at 600 °C improved the thermal characteristics of the hybrid composites [72]. Finally, bamboo fibers, polypropylene, and polylactic acid mix composites were created, and their morphological and thermal characteristics were compared to those of plain polymers [73]. Adamu et al. [74] studied the impact of polyvinyl alcohol/acrylonitrile on bamboo nanocomposite and found out that both clay and polymer act as filler materials having a complete dispersion as shown in Fig. 2a–e.

Bamboo fibers and highly hydrophobic polymers have poor interfacial interaction because of various functionalities, notably hydroxyl groups [75]. Researchers have attempted to enhance these qualities with various interfacial treatments [76]. It has been widely researched how different matrices, including polystyrene, polyester, and epoxy resins, affect the strength of bamboo fibers. Bamboo fibers' high economic worth, specific strength, low weight, and non-hazardous nature are among these materials' most desirable characteristics, motivating researchers to concentrate on composite technology. As a result, composites made with bamboo fiber have been used in the automobile industry. They have the potential to take the place of expensive, non-renewable synthetic fibers in a variety of industries, including the home. Many nations have recently passed rules requiring 95% recyclable materials in automobiles because of an ecological hazard. The current age demands the incorporation of natural fibers into daily life, especially composites made with bamboo fiber. Using these bamboo fibers in composite materials has thus become the subject of intensive research in many fields, including engineering, biotechnology (genetic engineering), farming, and others.

4.1 Bamboo Fibers and Its Composites' Physical and Mechanical Performance

Environmental worries about the depletion of fossil fuels and the increased awareness of plastic waste led to the development of biodegradable polymers. Biodegradable polymers are mostly made from renewable, recyclable, reasonably priced natural resources that are also ecologically benign [77, 78]. By using biodegradable materials, which can be readily disposed of owing to their microbial activity and assisting in lessening the issues of plastic waste, it is possible to alleviate the environmental challenges of synthetic plastics. Researchers and developers from a variety of industries have focused their attention on biodegradable polymers to date to replace common synthetic polymers [79]. Natural fibers have been bonded with biopolymer to increase the function of the material and provide sustainability. Biocomposites

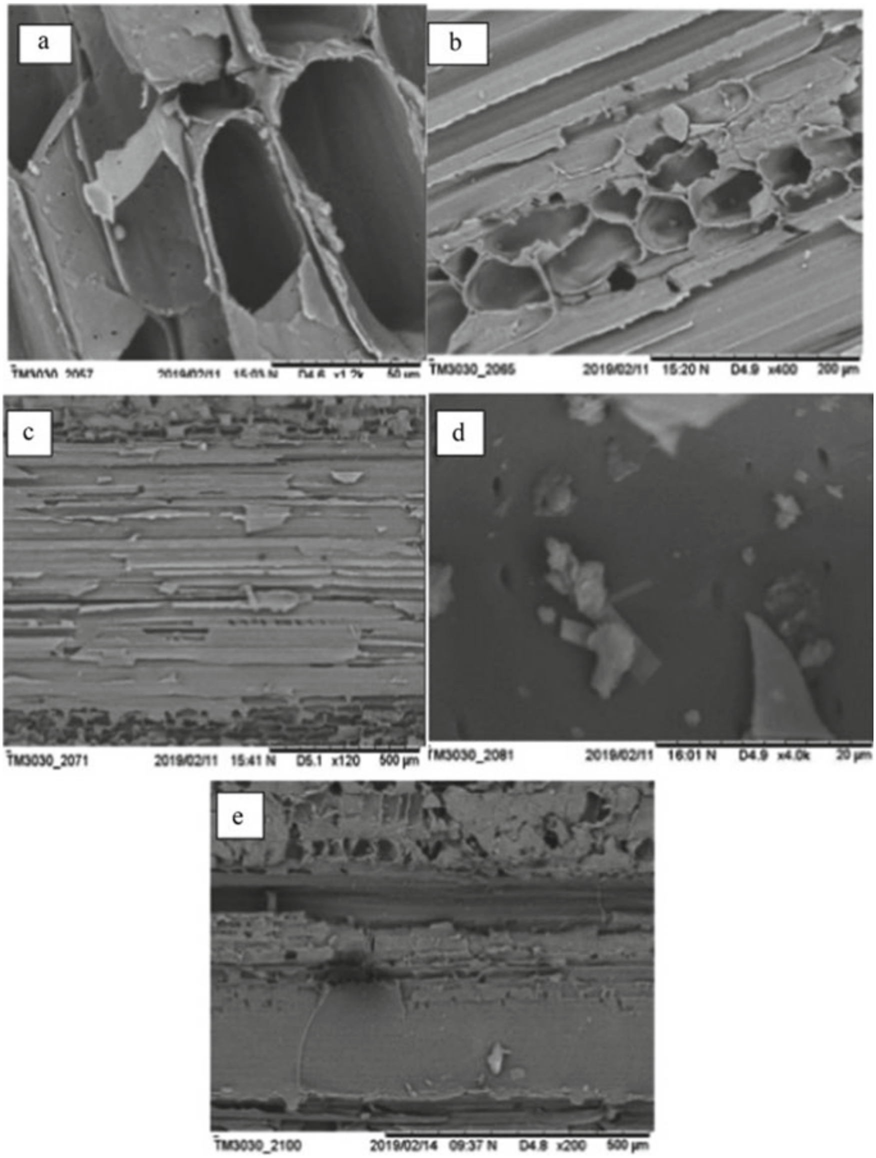

Fig. 2 SEM images show the **a** raw fiber, **b** bamboo nanocomposites 1 (BNC1), **c** BCN2, **d**, BNC3, and **e** BNC 4 [74]

are known to have high availability, full biodegradability, and economic viability [77]. Bamboo fiber is one such material that has enormous promise, particularly for polymer composites' reinforcing materials [80]. Bamboo fiber has been used progressively as reinforcement filler in biopolymers to improve mechanical performance and broaden their applications. In conclusion, using bamboo fiber as reinforcement in biodegradable polymer has outstanding potential to be extensively used in economic growth, especially in an industry in many sectors that need the application of eco-friendly products [41]. Furthermore, biodegradable polymers often reported improved mechanical performance after being reinforced with bamboo fibers, which may be related to various variables, including bamboo fiber treatment, length, and orientation.

The most well-known biopolymer is poly (lactic acid), produced from renewable agricultural raw materials and applied in various fields. Due to its remarkable mechanical qualities, high stiffness and strength, superior thermal capabilities, and biodegradability, PLA has been extensively employed to replace polymers made from fossil fuels [79]. It has been frequently mixed with inorganic fillers or natural fibers, such as wood, kenaf, flax, jute, ramie, and bamboo, to improve the properties of PLA and produce bio-based composites [81, 82]. Because of their superior physical characteristics, PLA composites with bamboo fiber reinforcements have been commonly used, particularly to improve their mechanical and thermal performances. For instance, Ochi et al. (2015) investigated the effects of adding bamboo fiber to a PLA composite on its mechanical properties. A steam explosion technique was used to extract bamboo fiber with a diameter of 100–300 m and then to make an emulsion-type PLA. Bamboo fiber content (0, 30, 50, and 70wt%) and molding temperature (120–200 °C) were adjusted to see how they affected the composites' mechanical performance, notably their flexural capabilities. The factors chosen for each sample and the composite's flexural properties were shown to have various characteristics throughout the investigation. The flexural strength plateaued between 120 and 160 °C and dramatically declined. At 160 °C, the flexural strength reached its maximum value. According to the optimal temperature, the influence of fiber content was determined, and it was discovered that the flexural strength increased linearly with increasing fiber content. Compared to the previous data, with 70 wt% bamboo fiber loading, the flexural strength was determined to have a greater value at 273 MPa. However, [78] used bamboo fiber to investigate how this reinforcement affected the PLA biocomposites' mechanical and creep resistance. With the addition of 60 wt% of bamboo fiber, the PLA composites had one of the best reported rupture and elasticity moduli. The characteristics, however, were shown to degrade when the fiber loading rose over 70% by weight.

A time-consuming approach was necessary to optimize the composites' production conditions to assess their mechanical performance. Nurul Fazita et al. [83] have used the Taguchi technique to obtain the best mechanical properties for the composites through achieving ideal production parameters by investigating the influence of the control parameters on the fiber-matrix interaction. First, the composites were created using a twin-screw extruder and a melt blending technique. They were then hot-pressed at 170 °C at a pressure of 81.58 kgf/cm^2. The Taguchi analysis shows that medium fiber sizes ranging between 150 and 250 mm, low fiber loadings of 10 wt.%, die temperature of 180 °C, and the screw speed of 200 rpm, give the maximum tensile and flexural strengths for the composites.

The orientation of the composites also has a significant impact on how well the composites system performs mechanically. Landes and Letcher [84] illustrated the influence of different orientations on the composites made from woven-bamboo mats reinforced PLA. In contrast to other orientations, the 0°/90° orientation had the best performance with the tensile strength and maximum elongation at 30 MPa and 30%, respectively. Further tests revealed that the change in orientations had little effect on the flexural properties, with 45°/45° and 0°/0° orientations portraying greater degrees of flexural strength than the 90°/90° and 0°/90° orientations. According to the shear test, every orientation of the composite exhibited brittle behavior. The investigation revealed that the PLA thermoplastic composites reinforced with bamboo mats had a good and acceptable result with few flaws and the substantial abilities that are very rarely seen in natural fiber reinforced thermoplastic composites. A different study by [85] observed even better tensile strength in PLA composites reinforced with bamboo fibers. Tensile strength increased by ~20% (29–35 MPa) with the addition of only about 5 wt.% of bamboo fiber.

Another study was carried out to produce a membrane film of PLA-reinforced bamboo fiber. Le Phuong et al. [86] researched membrane separation, which is currently a growing energy-efficient technology, using PLA biopolymer to create sustainable, biodegradable, nonwoven bio-based composite membrane supports with bamboo fiber and dimethyl carbonate. Bamboo fibers were bound together in the composite using PLA as a binder, resulting in a porous structure with a rough surface suitable for membrane support applications. The author found that the bio-based membrane support had a porosity of about 0.719 ± 0.132 and similar tensile strength to that of the synthetic polymer. Additionally, the composite film's mechanical stability is enhanced, swelling is reduced, and perforation is increased by up to $1068 \pm 32\,\mathrm{l\,m^{-2}\,h^{-1}\,bar^{-1}}$ in water. Furthermore, bamboo fiber's insoluble nature in most solvents also aids in enhancing the membrane supports' chemical stability. The membrane supports also showed to be durable in the long-run when it was exposed to continuous crossflow filtration for two weeks and submerged in water for more than six months. This finding demonstrated the porous properties of the bamboo-reinforced PLA composite films, which make it suitable for applications other than membrane support, such as microfiltration membranes and adsorbents.

Biodegradable polymers are often mixed with other polymers in many studies and utilized as different matrix systems in composites. The tactic of polymer mixing is thought to be particularly helpful in producing novel materials with the best possible structure and performance. This multi-component hybrid system still has a lot of promise due to its exceptional mechanical and processing capabilities. For instance, Long et al. [87] used PLA biopolymer and a synthetic polypropylene (PP), as the two different matrices, while reinforcing them with bamboo fiber to investigate the blending process. The bamboo fiber's surface had undergone various modifications, including using coupling agents like MAPP to improve the matrix and fiber compatibility. The combination of the matrices and modified bamboo fibers were used to create samples using a twin-screw extruder and 3D printing with the Fused Deposition Modeling (FDM). The investigation discovered that altering bamboo fibers to remove hemicellulose and pectin constituents and reduce their hydroxyl content improved the interfacial adhesion between PP/PLA and bamboo fiber. Furthermore, the inclusion of 5% MAPP in the BF/PP/PLA hybrid composites was found to be the ideal composition with best tensile, flexural and impact strengths that increased by 13, 11.7, and 23.5%, respectively, compared to the neat BF/PP/PLA composites. Due to their better qualities, BF/PP/PLA hybrid composites are a viable substitute for pure PLA, particularly for use in 3D printing.

Besides PLA, only a few different biodegradable polymers have been employed as the matrix for fabricating composites, especially with bamboo fibers. Poly (butylene succinate), more commonly known as PBS, forms one of those rare biodegradable and environmentally friendly polymers, which is generally produced from biowastes. It consists of 1,4 butanediol and succinic acid. Pivsa-Art and Pivsa-Art [80] researched reinforcing bamboo fiber in PBS biopolymer. They studied the effects of varying fiber matrix composition on the biocomposite's mechanical and physical properties, which can be used in indoor furniture components and in packaging as well. PBS has historically been used because of its great qualities, including good biodegradability and strong toughness owing to its low glass temperature. Alkaline-treated and untreated bamboo fiber of two distinct kinds were employed at various loadings (10, 20, and 30 wt%). This investigation discovered that alkaline treatment showed greater tensile strength than the untreated ones. When PBS is reinforced with 30 wt.% bamboo fiber, the composites' Young's modulus rises to 2575 MPa compared to only 639 MPa for pure PBS. Results for Izod impact strength also showed similar pattern, proving that alkaline treatment is beneficial in removing lignin and hemicellulose elements from the bamboo fibers and improving the surface interface between matrix and fiber. For composites to be used in industry, appropriate performance, especially mechanical qualities, is essential. Furthermore, [88] have examined PBS-based composites reinforced with nano/micro hybrid bamboo fibers, with the aim of creating useful fiber-based composites with superior strengths. The mechanical performance of the hybrid composites was observed to be greatly enhanced using hybrid nano- and micro-scale bamboo fibers. The best tensile strength and Young's modulus were reportedly achieved when a combination of carbon nanofiber (CNF)/BF, which had been thermo-compressed for 15 min, was added to PBS, increasing these properties by 240 and 700%, respectively, over neat PBS.

Another topic was the inclusion of bamboo fiber in naturally occurring materials formed from biodegradable polymers, such as starch, seaweed, etc., which was also investigated. For example, [89] used biopolymer film made from red seaweed reinforced with bamboo fiber (*Kappaphycus alvarezii*). First, red seaweed was used to extract the carrageenan, treated with a solution of 1 M NaOH. After being neutralized with HCl, the mixture is dried and processed as a biocomposite film. In contrast, the bamboo fiber was also converted into cellulose pulp before being combined with red seaweed carrageenan to produce biocomposite film. Before being cast, the seaweed solution was mixed with several loadings of bamboo pulp (0, 1, 5, 10, 15, and 20%). The analysis shows that the mechanical properties of the seaweed matrix were improved by the addition of bamboo pulp fiber, with tensile strength being 109.1 MPa, Young's modulus being 55.4 GPa, and improvement in stretchability before breakage being 22.3% in comparison to control. Furthermore, after the biopolymer was combined with bamboo fiber, improvements in water vapor permeability qualities and a high contact angle of 91° were also noted. The possibility of using biodegradable biopolymer composite films as packaging materials, particularly for the food industry, was confirmed by this discovery.

Yusof et al. [90] used bamboo fiber to strengthen the natural polymer composites made from tapioca starch and PVA to compete with the market's standard petroleum-based plastics. It has been claimed that subjecting the composites to various forms of chemical pre-treatment helps to boost their tensile and flexural strength. There in the study, treatments with permanganate and alkali were carried out. The composites were made by solution casting a mixture of starch, PVA, and bamboo fiber using glycerol as a plasticizer. The composites comprising of bamboo fibers treated with alkaline solution had higher mechanical performance in comparison to those with untreated and permanganate-treated fibers. According to the author, adding bamboo fiber improved the biocomposites' heap bearing limit and ability to withstand bending, which improved their tensile characteristics. In addition, removing undesirable ingredients from the fiber cells during the fiber treatment further improved the composite sample's mechanical characteristics. It enhanced the interlocking between the fiber and matrix. While the hybrid biocomposites' flexural characteristics also exhibited a similar pattern. The alkaline-treated sample performed the best since it was hardly breakable, indicating great flexural strength. Even though all samples had fibers of an equal length, this occurrence followed the sample's stress–strain relationship, as seen by its tensile strain behavior, determined by the bonding strength between the fiber and matrix.

In a separate study, [91] investigated the possibility of combining a hybrid polymer, made by mixing starch and PVA, with nanocelluloses that are extracted from bamboo fibers. The addition of only about 6.5% nanofibrils affected the tensile strength (increase of 24%) and elongation at the break (increase of 51%) in the composites. Furthermore, the tensile properties of the small-fiber reinforced composites were substantially superior to those of the bamboo fiber reinforced starch/PVA composites, reported earlier.

4.2 Bamboo Fibers and Its Composites' Thermal and Fire Performance

Although many publications on the characterization of composites made from natural plant fibers have been thoroughly investigated, reports on the fire and thermal characterization of composites made from fibers, especially bamboo, are rare. Das et al. [92] fabricated a composite with novalic resin reinforced by bamboo fibers that were again treated with alkali. The thermogravimetric analysis helped establish superior thermal stability of the fabricated composites. The novalic resin functioned as a glue that was responsible for attaining excellent bond between the constituents, resulting in higher bonding forces and superior thermal stability. The affinity of fibers for absorbing water reduces after alkali treatment because corresponding soft metal ions occupy most of the external hydroxy groups, resulting in neutralization that causes new connections between the inherent cellulosic molecules. The creation of these bonds results in a firmly closed-packed structure of cellulose, which raises the bamboo treated with an alkali crystallinity index and increases stability [93]. This caged system supports the water of crystallization, increasing the temperature at the end. After being treated with alkali, bamboo fiber-based composites' thermal behavior, such as heat deflection temperature, was observed and claimed to have somewhat improved [94–97]. Despite a minor increase in thermal stabilities, the thermal deterioration pattern changed after fiber was added. After adding fibers, the composites' deterioration pattern changed, and their thermal stabilities somewhat improved. A significant factor that directly affects a material's thermal behavior is the fiber composition of the composite. Researchers highlighted that partial deterioration is a good cause that directly influences the thermal stability of bamboo composites in one of the comparative biodegradation studies [94]. It has been described how the structural characteristics of bamboo composites relate to different characteristics, including crystallization and interfacial morphology [98]. DSC curves were studied to evaluate the influence of the neat matrix and their alternatives. It was observed that the modified matrix had a significant influence on the DSC curves, which can be attributed to the presence of an adequate amount of b-phase form.

According to some studies, the kind of filler employed impacts the qualities of composites; for example, inorganic fillers have the potential to operate as b-nucleators and generate significant amounts of b-form, which directly impacts the thermal characteristics of the composites [99, 100]. In another investigation, bamboo fiber was considered a potential source of b-nucleators [98]. Stronger adherence between the matrix and bamboo fibers, which results in improved thermal stability, is the root of the varying degrees of thermal behavior. It tested how modified bamboo-reinforced composites with various fiber loading levels responded to heat [101]. The thermal behavior of bamboo, neat matrix and the resulting bamboo composites were compared. Increased inorganic silane concentration on the bamboo surface was found to impact maximum peak temperature directly and directly correlate with weight loss. According to the studies on the polypropylene-reinforced bamboo fiber composites' kinetics, the peak of crystallization and the starting temperatures reduced with increased cooling rate [102]. It was explained that slower cooling rates provide polymer nuclei enough time to reach an active state, which causes crystallization at higher temperatures [103]. The peak of the crystallization temperature at a specific rate of cooling had direct relation to the amount of bamboo fibers used, a finding that helped substantiate the heterogeneous "nucleation effect" of bamboo [104–106]. The studies achieved in getting lower crystallinity in the PP-BF composite, proving that a slower cooling rate was required [107–109].

The matrix melting temperature was reported to be somewhat reduced, and crystallization was enhanced when bamboo fibers were added to recycled polypropylene-based bamboo composites, indicating that the quantity of bamboo fibers directly influences the thermal characteristic of composites [102]. Other studies have also shown that recycled polypropylene crystallization rates and crystallinity percentages increased [110–112]. Bamboo fiber, when used in various loading percentages, positively affects the recycled resins' crystallization that frequently occurs at higher temperatures. The primary reason for this behavior is the nucleation effect [113, 114].

Guo et al. [115] method successfully converted natural bamboo into a composite with superior performance and having lower weight, higher tensile strength, and limited flammability. Natural bamboo is first de-lignified, woven in a superior aligned formation, and are treated before being injected with epoxy resin. The hydroxyl groups of boric acid (BA) and the cellulose backbone's diol or carboxylic groups cross-linked with each other, forming a stable and strong compound, when BA was applied in an alkaline solution. Due to the well-preserved aligned bamboo fibers, the BA-bamboo/epoxy composite showed increased tensile (by ~234%) and impact strengths (by ~177%), compared to pure epoxy. The BA-treated composite had comparable mechanical properties to the untreated ones but had substantially improved flame retardancy abilities. The treated bamboo/epoxy composite's limiting oxygen index was '26.5% larger, and cone calorimeter studies proved that the peak heat release rate (PHRR) was also ~63% lower than the original epoxy resin. Furthermore, BA resulted in greater charring of the composite, which was also proven in the thermogravimetric analysis with lesser mass loss and higher thermal stability, resulting in the substance's considerably improved fire performance. The properties of the treated composite had excellent performance to be able to be used as structural materials.

Flame-retardant bamboo-PP composites were produced by melt blending, with ammonium polyphosphate (APP) as the flame retardant and bamboo flour as the smoke suppressant and investigated by [116]. When employed as a synergist, bamboo flour can greatly improve the smoke suppression and flame retardancy performance of PP/APP composites, which was prominent in the study. It also helped improve the composites' mechanical performance, thermal degradation, and crystallization behavior. Therefore, various characterization techniques were employed, including scanning electron microscopy, cone calorimetry, thermogravimetric analysis, and limiting oxygen index, to investigate the microscopic morphology of carbon residue, flame retardancy, thermal stability, and combustion characteristics. According to experimental findings, the PP55/APP30/BF15 composite had the greatest smoke-suppression effect when APP/BF was mixed in 2:1 ratio, amounting to about 45 wt.% of the composite. It's because the residual carbon has more micropores and surface area, resulting in reduced PHRR to 308.2 kW/m^2, and increased carbon residual of 25%. In addition, the continuous carbon layer created during combustion may shield the matrix material and stop heat transmission.

Chen et al. [117] examined the fire performance of designed bamboo beams when exposed to three-sided standard fire conditions. Nine tests were carried out, including two fire tests with a continuous applied load and the remaining flexural tests on the manufactured beams when exposed to the standard fire conditions. The reported parameters included the load ratio in fire tests, the duration of the fire exposure, the shaft's geometry, and the type of bamboo used to design the test conditions. Two products made from engineered bamboo frequently used in construction are bamboo scrimbers and laminated bamboo structures, which were also tested in the study. In every case, regardless of exposure to fire, the failure mechanism was flexural coupled with tensile fracture under extreme tension. Longer fire exposure times reduced engineered bamboo beams' stiffness and maximum loads, but the strain distribution along the middle span of the structures' cross-sections remained linear. As the applied load increased, the fire resistance of laminate bamboo beams decreased. After testing, each beam's charring depth was determined, and an equivalent charring rate was computed. The findings indicated that the laminated bamboo beams were charred more quickly than the denser bamboo scrimber beams. Sectional analysis and the reduced cross-section approach were employed to calculate the structures' resistance to fire and loading capacities.

5 Applications

In recent years, efforts to use bamboo as a renewable non-wood fiber have been strengthened. This renewable fiber has improved agroforestry since bamboo grows fully and matures in one and two years. Theoretical and practical research on products made of bamboo has evolved, notably in furniture, packaging, building, and transportation, since bamboo grows swiftly and is renewable. Composite materials have supplanted both interior and outdoor applications of traditional wood. When their strengths were compared, they were found to be substantially stronger (~10 times) than normal wood produced. Bamboo composite products generally have advantages over conventional composites and traditional wood due to their low maintenance, weather resistance, dimensional stability, longevity, high impact resistance, and restricted flame spread.

Today, fences, decking, deck tiles, dustbins, railings, outdoor furniture, and decking accessories are all made from bamboo composites, which usually contain up to 70% bamboo fibers and roughly 30% high-density polyethylene (HDPE), making the entire composite recyclable. These bamboo composites were designed specifically to maintain their shape and stability, be resistant to termites, have excellent stability under varying thermal conditions, and be easy to handle, transport and install. These composites made from bamboo provide enough cost and benefit to the end consumers. For structural purposes, laminated bamboo lumber (LBL) has been published in recent publications [118]. Despite the success of the LBL fabrication effort, more must be learned about how changes in strength qualities affect the LBL. The current bamboo-based composite industry has significantly influenced the development of modern economical techniques, which are less reliant on the commonly used renewable building material, wood, while being eco-friendly, producing reduced pollution, and consuming less energy. Outdoor constructions in the building industry,

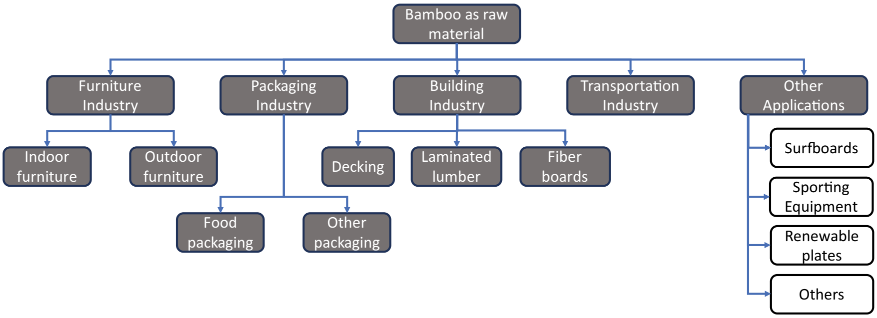

Fig. 3 The various applications of bamboo and its by-products

such as decking, form another use for bamboo composites. These decks are made of multiple layers of bamboo boards coated with epoxy matrix and a special coating over glass boards to prevent bending and preserve their shape over time. Surfboards are a general term for water-surfing composites made of bamboo. These surfboards are lightweight, have a distinctive shape, and have water-resistant surfaces. Recently, Mitsubishi Japan created a prototype using composite materials based on bamboo. They made this prototype using polyurethane resin. By employing plant-based materials, Mitsubishi could minimize the cost of manufacturing the present materials and the CO_2 emissions. Advanced composites are seldom the first choice for materials utilized in automotive applications, despite their potential advantages. Advanced composites require a significant shift in economic thinking, and the only solution is using natural plant fibers. Bamboo is a great resource to use in place of the current commonly used synthetic ones, especially with the growing raw material shortages and price spikes. The vast application field of bamboo as a raw material is illustrated in Fig. 3.

6 Conclusion and Future Perspectives

Using bamboo fibers in various products has created new opportunities for academics and businesses to develop a sustainable module for using bamboo fibers in the future. Bamboo fibers are widely employed in composite industries to help people advance their socio-economic status. Bamboo market values are strongly impacted by developing cost-efficient and environmentally friendly biocomposites made from bamboo fiber-based composites made from various matrices. Detailed research of bamboo fibers' mechanical, physical, and basic characteristics is required to build such composites. As a result, this review has attempted to compile data on the fundamental attributes of composites manufactured with bamboo fiber and their practical applications. Researchers from around the globe have conducted a broad variety of research using cutting-edge ideas to provide working and employing communities with essential assistance. Current research on composites based on bamboo fibers uses both basic and practical science. Even though other countries like India and China have made significant progress in the socioeconomic use of bamboo fiber, the ultimate goal of fully utilizing bamboo fiber is far behind its anticipated milestone, particularly in Malaysia and other countries. It would be possible to use bamboo in ways other than the typical conventional mode with the support of the industry's sustainable future for bamboo-based composites. The efficient characterization of bamboo fiber and composites made from bamboo fiber should be furthered in terms of research and testing. Even though scientists have done much work on composite materials made from bamboo, more study and innovation are still needed to address potential issues. These things will benefit urban and rural residents who depend more on composites made of synthetic materials.

A sustainable future depends on the current industrial development towards the economical and efficient production of the industrial goods and their eco-friendly production process. The market's dominance of synthetic/petroleum-based goods can be challenged by the biodegradable and renewable plant materials, only if they are high-performing, depleting petroleum usage and building a new foundation for environmentally friendly and sustainable products. Natural fibers and biocomposites made from natural resources, if can possibly be incorporated into sustainable and well-designed industrial products, have the potential to challenge the dominance of products derived from petroleum. Bamboo fiber is well-known for its renewability in terms of rapid growth and outstanding mechanical properties. Using cutting-edge technology, using bamboo fiber to create biocomposites transforms the next generation's future. A new revolution in resource conservation may be sparked by carefully crafted goods made of bamboo fibers. This quick evaluation suggests that bamboo fibers can create sophisticated, engineered products for various uses. It will be a different approach to creating biocomposites, particularly those used for common people's daily needs, such as lightweight automotive parts, sports equipment, fences, decking, and flooring. Their affordable prices, simple accessibility, and appealing designs will be the primary impetus for transitioning from a dependent present to a sustainable future.

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Effect of Interfacial Bonding Characteristics on Fire Performance of Flax Fiber Reinforced Composites

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Abstract Flax fibers are mainly oriented to their use in handicrafts, furniture, decorations, carpets. However, in recent years, these fibers have been directed towards other industrialized sectors such as automotive, construction, military, and transportation due to their high rigidity and resistance. Flax fiber is often used as a reinforcing phase in thermoplastic composites and, to a lesser extent, as a natural reinforcement in thermosetting resins. In this way, composite materials reinforced with flax fiber are in the spotlight for developing new products and boosting other markets, complying with environmental regulations, and reusing abundant waste at a low cost. However, natural fibers have specific weaknesses, such as their hygroscopic nature and low resistance to fire, and flax fiber is no exception. Additionally, the intrinsic nature of the constituents of the composites (linen fiber and polymer resins) are generally incompatible, so the interfacial bonding is fundamental to looking for optimal mechanical performance and its behavior against fire, essential for its use and commercial application. Optimizing the interface between the natural reinforcement and the polymeric matrix is perhaps the most critical aspect in developing polymeric composites reinforced with flax fibers. Natural fibers are flammable and therefore add to the flammability of composites in which the organic matrix is also volatile. This book chapter presents an overview of the effect of interfacial bond compatibility between components of natural fiber-reinforced composites, including surface treatments intended to improve compatibility and strategies to increase the fire resistance of flax fibers.

Keywords Natural fibers reinforced polymers · Interfacial bonding · Flax fiber composites · Biobased composites · Fire performance of composites

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1 Introduction

Long fiber-Reinforced Plastics (FRP) are composites where fillers and additives are added to the polymer resin to increase the mechanical properties, provide multiple functionalities and reduce manufacturing costs. It is widely known that the reinforcing phase, the fibers, determines the stiffness and resistance of the composites since it supports the applied loads. For its part, the polymeric resin protects the fibers and transfers the stresses between them. The polymer matrix determines the maximum service temperature and can be thermosetting or thermoplastic. Thermoplastics are semicrystalline or amorphous polymers that can be reprocessed multiple times, leading to chain scission degradation. Thermosets are generally liquid with low viscosity. The monomers are joined by the addition of an initiator and activator, which produce an exothermic reaction during the formation of a three-dimensional network that cannot be broken down or melted again.

Epoxy resins are thermosetting polymers extensively used to impregnate carbon fibers, so their use is mainly focused on manufacturing composites with structural applications. Polyester resins are commonly used in glass fiber-reinforced composites.

A wide range of plastics and fibers allows the development of various composite materials with diverse applications. However, the consulted literature exposes the results of just a few fiber-polymer configurations [1–10]. Figure 1 presents the conventional classification of composites based on the configuration and arrangement of the reinforcement phase [1–10].

The long fibers are used to reinforce polymers but are also used in manufacturing textile products. The latter extensively uses thermoplastic fibers, commonly polyester or nylon fibers. For their part, synthetic glass and carbon fibers are used in structural applications and engineering components. Manufacturing these synthetic fibers involves high energy consumption, health impacts, environmental changes, expensive production costs, and a depletion rate of oil supply, justifying the search

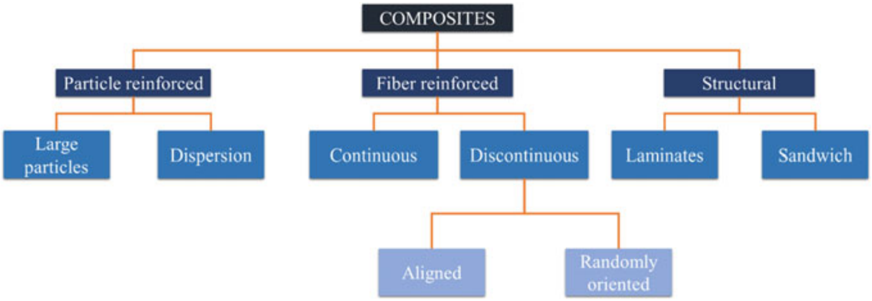

Fig. 1 Conventional classification of composite materials based on the reinforcement phase, adapted with permission [10] provided by Springer Natureand Copyright Clearance Center

for affordable alternatives for FRP composite manufacturing that are less expensive and friendly to the environment.

An ecological alternative is using natural fibers, which are renewable, biodegradable, low density, and have acceptable mechanical performance [11–13]. Despite these advantages, natural fibers have some limitations, such as their high hydrophilicity, which weakens the natural fibers/polymer matrix interface. Interface bonding is the boundary between the fibers and the polymer matrix through which loads are transferred. If the interface fails prematurely, load transfer cannot occur, and the fibers' reinforcing effect is limited.

This chapter deals mainly with the interface and bonding mechanisms of natural fiber-reinforced polymer composites, primarily focusing on the compatibility between the constituents, the modifications and the improved compatibility, the bonding mechanisms, and the composite's interface structure.

2 Natural Fibers

Today, the need for industrial products that are friendly to the environment is growing strongly because of ecological concerns, environmental care, and international regulations. Using natural fibers as a reinforcing phase in composites has great potential since they offer relevant thermal properties, have low density, and are not expensive. These competitive advantages allow the industry to consider natural fibers a viable alternative in manufacturing environmentally friendly composites [14–16]. Although the processes for obtaining natural fibers are not toxic and do not affect workers' health, they cannot yet be used for structural components because of their limited mechanical performance.

Natural fibers are classified as plant fibers, which contain cellulose, and animal fibers, made up of proteins, so they are not used as fillers in composite materials. Figure 2 [9, 10] presents the general classification of natural fibers.

Natural fibers are primarily composed of cellulose (the main component), lignin, and hemicellulose [17–22]. In more detail, some investigations raise additional compositions, such as pectin, a polysaccharide starch of low molecular weight that acts as gelling agent [23].

Figure 3 presents the cellular structure of plant fibers [24]. The microstructure of natural fibers is made up of the lumen surrounded by a cell wall. Cell walls contain primary and secondary microfibrils of oriented semicrystalline cellulose in an amorphous hemicellulose-lignin matrix [14, 19, 21].

Flax fiber

Flax fiber (Linum usitatissimum) belongs to the liber family. The first records of the use of this material date back to prehistory. Flax fibers had their heyday in the Middle Ages, although they did not displace cotton fiber, which was mainly used for clothing and common textiles. Even so, flax fiber has become one of the most used and sustainable natural fibers. Flax fiber is the most relevant plant fibers due to its

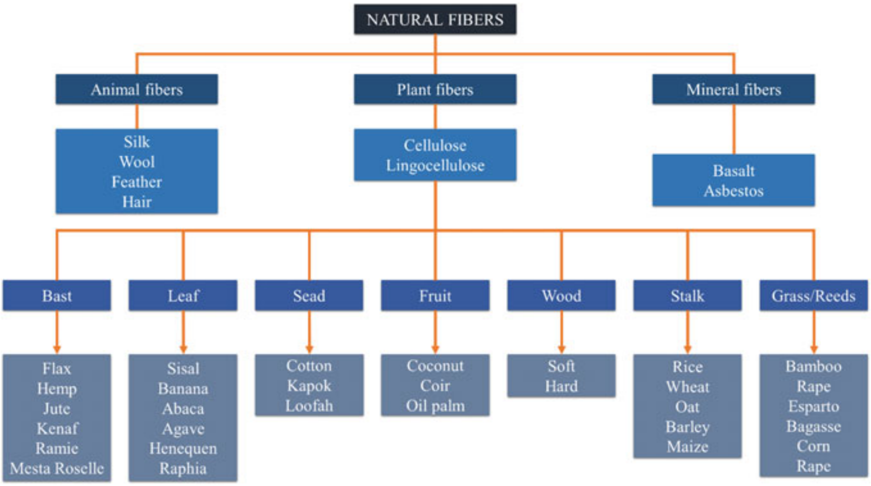

Fig. 2 General classification of natural fibers, adapted with permission [10] provided by Springer Natureand Copyright Clearance Center

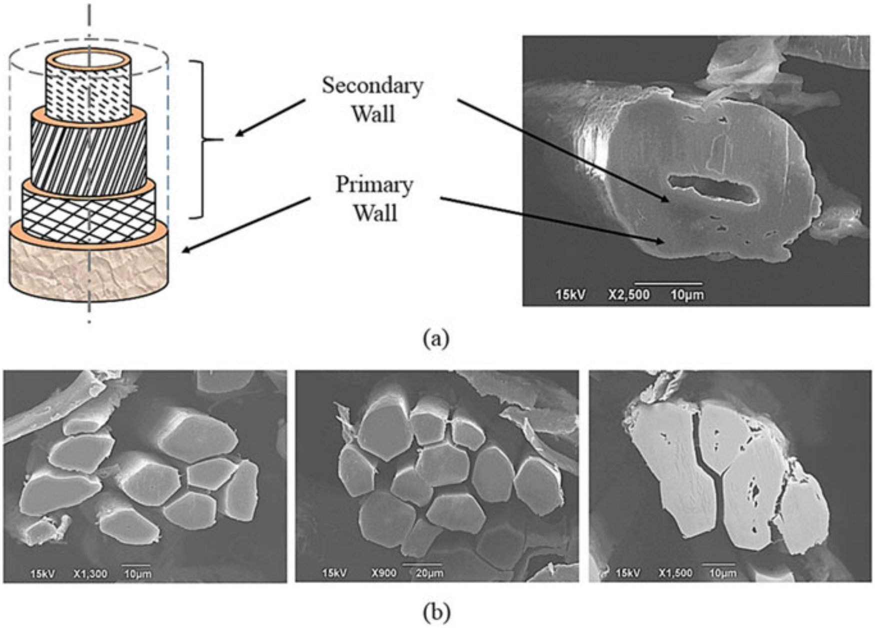

Fig. 3 Plant fiber microstrucure. Reprinted from [25] open access Creative Common CC BY license

intrinsic properties. These fibers, for example, are resistant to UV rays and have good mechanical properties, which gives them durability in composite materials and is easy to grow. The main growing areas for textile flax used to be in Western Europe (France, Belgium, the Netherlands, and Germany). However, China has become the largest exporter of this fiber, leaving the European community far behind. Flax fabric is the oldest fiber of humankind. Man's interaction with this material began in prehistory. In ancient times, flax fiber was used to make clothing. In some documentaries, it is mentioned that flax fabrics were used for packaging the deceased. The ancient Greeks used flax to make clothing and household items, but the Phoenicians used it for their ships' sails and expanded its reach to the rest of the world. Sometime later, its use branched out, and they began to use it in painting as a canvas; it was also used to make writing paper for more than seven centuries.

The heyday of flax weaving in Europe during the Middle Ages lasted several centuries. Only wool was more relevant than linen, commercially reaching the American continent. After the industrial revolution, linen was displaced because it could not compete with low prices. Then, the textile industries began to produce flax fiber mixed with other fibers, such as cotton and viscose, to reduce costs.

Despite today's technological advances, linen fabric continues to have a production process related to craftsmanship. In addition, the flax plant requires excellent attention, and since the fiber is not elastic, it is not easy to weave without breaking the threads.

Currently, the flax plant is cultivated in regions with a temperate climate [25]. One characteristic that stands out about flax is that it is organically grown: it is planted naturally, without the need for fertilizers or pesticides, and it hardly needs watering during the growing months. Likewise, processing and transforming it into tissue does not require much energy; it does not generate waste and is biodegradable. Like the rest of the plant fibers, the mechanical performance of the flax fiber depends on climatic conditions, which favor its cultivation and alter the plant's growth. The growth of the flax plant is of particular interest since the flax fiber's mechanical properties vary along the plant's stem [26]. As mentioned above, China is currently the largest producer of flax. However, high-quality production is still an essential part of the cultures of many European countries. Also, the fabrics of the United States and India are recognized for their quality. In those markets, more work is done in upholstery and household items.

The mechanical behavior of composites containing flax fibers are subject to the extraction process and the area of the stem from which the fibers are obtained. The middle zone of the plant includes flax fibers with superior mechanical properties than the lower and upper zones of the stem [27, 28].

Manual or automated processes extract flax fibers. Hand-drawn fibers have superior mechanical properties over mechanically drawn fibers because automated drawing processes twist them, reducing their strength. Therefore, the mechanical parameter values of manually extracted fibers are widely scattered compared to mechanically extracted fibers.

Additionally, it is possible to separate the natural fibers through enzymatic treatments. Zeng et al. [29] evaluated the mechanical properties of flax fibers by comparing the extraction process. The authors used an automated extraction process and a method for extracting flax fibers from an ammonia pretreatment. The mechanical parameters of tension and bending increased due to the binding applied to the flax fibers.

3 Interfacial Bonding Characteristics

As stated in the introduction, composite material's mechanical performance depends on the adhesion's quality between the two constituents [23, 28, 30], especially at the natural fiber/polymer matrix boundary (Fig. 4).

Various research articles and reviews present numerous strategies to modify the plant fibers' surface. According to Aswa et al. [31], the fiber-matrix separation starts with pockets developed on the fiber surface due to leaching from the fiber surface, as summarized in Fig. 5.

The flax fiber/polymer matrix boundary is a region where the reinforcement and matrix react to combine physically, mechanically, or chemically [25]. As previously commented, in the region of the interfacial union, chemical reactions occur where molecular forces allow mechanical interlocking, thus varying the properties of the composites. The molecular modification allows the hygroscopicity of plant fibers to be compatible with non-polar polymers. The hydroxyl groups contained in cellulose and lignin [32, 33] exhibit highly hydrophilic properties, which complicates the adhesion since most polymers are hydrophobic in nature [32, 33]. Therefore, the modification is critical to developing a molecularly compatible composite where the interface allows effective stress transfer, significantly improving the properties of the composite.

It is necessary to increase the affinity between flax fibers and polymers to improve the performance of the composites. The surface treatment limits the OH groups, leading to better adhesion between the natural fiber and the polymeric resin [23].

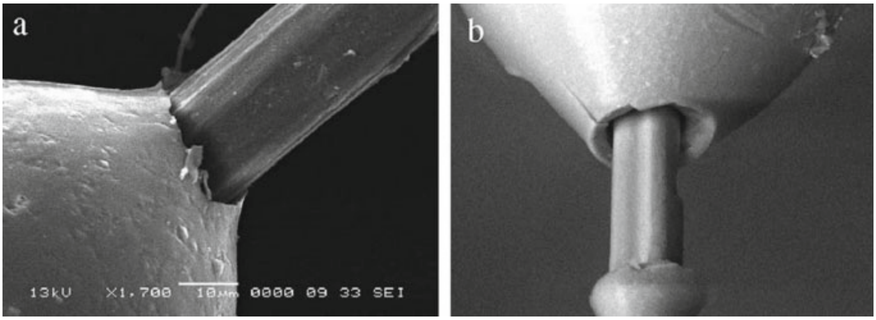

Fig. 4 SEM micrographs corresponding to **a** PLA and **b** epoxy, with permission [30] provided by Elsevier and Copyright ClearanceCenter

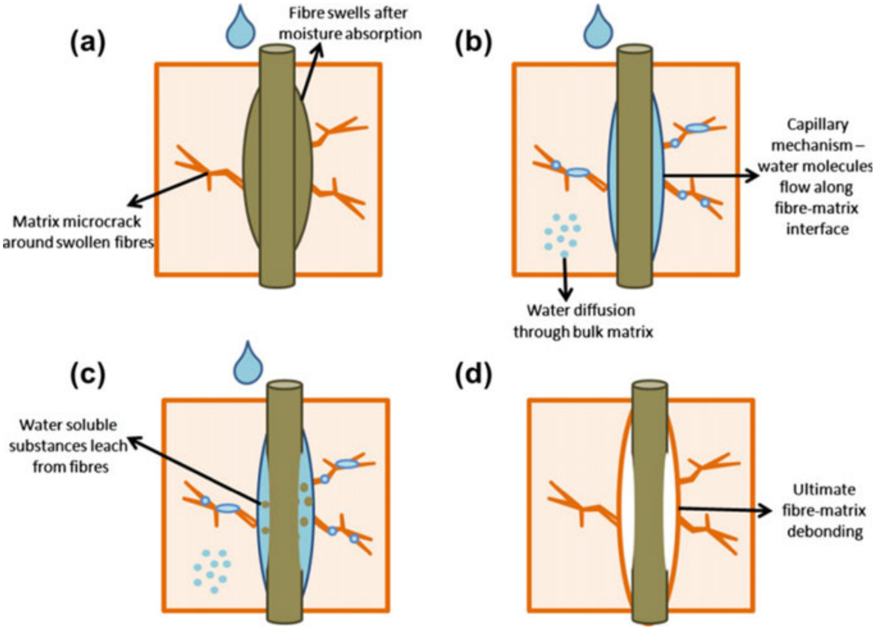

Fig. 5 Schematic representation of interfacial adhesion in the presence of water, with permission [31] provided by Elsevier and Copyright Clearance Center

Various physical and physicochemical treatments and chemical modifications have been evaluated to improve interfacial bonding between natural fibers and non-polar resins [34–41]. The growing environmental awareness worldwide encourages the research and development of biobased and sustainable plant fiber compounds. Polylactic acid (PLA) is one of the biobased matrices commonly used for biobased composite products, mainly due to the intrinsic polarity in its polymer chain, which provides better interfacial compatibility and adhesion with natural fibers [42].

This chapter will emphasize that the performance and properties of natural fiber composites depend mainly on three relevant phases: reinforcement, polymer matrix, and interface. The interface is often considered a phase transition that governs the matrix and the fiber junction. Shamsiah et al. [43] found that depending on the adhesion, the interface controls the tensional movements and provides the rigidity of the compound without damaging its structural integrity. The ability to move depends on the communication of molecules at the interface. Jiang et al. [43] performed a microscopic study in which the fibers pulled out excellently in both tensile and impact tests. The removal of the fiber suggests insufficient interfacial adhesion between the fiber and the matrix, as seen in Fig. 6. The coupling agent improved the interfacial adhesion, which increased the elastic modulus and strength values considerably. In addition, a significant energy dissipation during impact was detected, which substantially increased the impact resistance in composite materials [23].

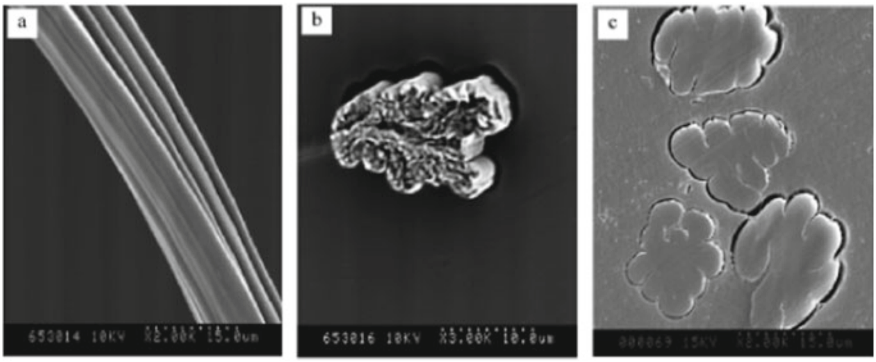

Fig. 6 SEM micrographs of bamboo pulp fiber surface. Reprinted with permission from [43]. Copyright 2022 American Chemical Society

4 Compatibility Between Flax Fiber and Polymers

Plant fibers comprise cellulose, often combined with other components such as lignin and other low molecular weight substances. As previously described, cellulose provides structural characteristics and is made up of crystalline microfibrils aligned with many hydroxyl groups. The hemicellulose acts as a binder for the cellulose microfibrils. Unlike cellulose, which has a crystalline structure and is resistant to hydrolysis, hemicellulose has an amorphous structure, low-strength, and hygroscopic nature so that it can be easily hydrolyzed with acids and bases. For its part, lignin is an organic polymer that provides rigidity and resistance to the flax plant. Therefore, the molecular structure promotes the hydrophilicity of flax fibers and leads to a lack of compatibility and adhesion between the fiber and the non-polar polymer matrix. The hygroscopic nature of flax fibers can favor hydrolysis during the manufacturing process of composite materials, either in the curing of the resin (in the case of thermosetting resins) or in the transformation (in the case of thermoplastic polymers). Various studies on the properties of flax fiber composites have been carried out, finding that flax fiber significantly impacts fire behavior and resistance to temperature in service. However, the flax fiber tends to agglomerate, making it difficult to distribute it homogeneously within the [44–50]. For these reasons, some research topics deal with physical and chemical modifications to reduce the hydrophilicity of the natural fiber to promote interfacial adhesion.

The physical treatments of the natural fiber alter the structure and the properties of the fibers' surface without requiring chemical components, enhancing the mechanical bond with the polymeric matrix, and improving the mechanical performance of the composite.

Ionizing radiation is a high-energy gamma radiation used to increase the mechanical properties of compounds through physical reactions or the reorganization of electrons around a nucleus. Gamma radiation deposits energy in the natural fiber, producing radicals in the cellulose chain through the egress of hydrogen atoms and hydroxyl groups, breaking carbon–carbon bonds and chain scission [23, 51, 52].

Motaleb et al. [53] investigated gamma radiation's effects on the different properties of composite materials. They found that the increase in dose altered the elongation capability.

Khan et al. [54] found that intermediate doses of gamma radiation increase the mechanical properties of PP composites reinforced with flax fiber. The authors measured the water absorption of the compounds at 25 °C and found that the treated samples had lower water absorption properties. Additionally, they found that the dielectric constant was higher for the treated composites than the untreated ones, reducing the composites' conductivity. Machnowski et al. [55] evaluated the effect of gamma radiation on the mechanical properties and surface structure of woven fabrics made of cotton, flax, and silk fibers. They found that small radiation doses cause negligible changes in fiber parameters, but higher doses cause considerable weakening.

Xia et al. [55] developed PLA-triallyl isocyanurate/pretreated flax fiber composites. Triallyl isocyanurate was used as the crosslinking agent. The treatment of the flax fibers consisted of a melt compounding process followed by gamma irradiation (0–20 kg). The authors found that the crosslinking agent favors the formation of a three-dimensional network of PLA during gamma irradiation, so the irradiated compound could only swell rather than fully dissolve in chloroform. Furthermore, irradiation improved the fire resistance and dimensional stability of the composites.

Radiation-induced graft polymerization allows modifying natural fibers conferring new surface properties and chemical reactivity with several polymers [56]. LeMoigne et al. [57] reviewed the impact of fiber irradiation on the properties of biocomposites. They presented several advantages of radiation grafting as an innovative strategy to functionalize natural fibers and improve the functional properties of biocomposites. Han et al. [58] studied the effect of the surface of natural fibers treated with electron beam irradiation (EBI) at various doses, noting that the interfacial strength depends mainly on the level of EBI treatment on the fiber surface. Their study suggests that proper modification of natural fiber surfaces at an optimal EBI dose improves biocomposites' interfacial properties. Mahzan et al. [56] explored the degradation of composite materials due to UV radiation, which carries a degradation rate due to different parameters such as wavelength, intensity, and exposure time. Their review highlights the influence of these parameters to provide a better solution for developing UV-treated polymer matrix composites. Yan et al. [27] evaluated the effect of UV radiation and water spray on the general properties of the composite reinforced with flax fabrics to evaluate its use in civil engineering. According to the SEM observations, the authors found a significant reduction in the mechanical properties of the analyzed composite materials attributed to the degradation of the fiber/matrix interfacial bond after exposure.

In general, the consulted bibliography concludes that at low irradiation doses, the elimination of hydrophobic compounds from the fiber surface can be counteracted by crosslinking some components of carbohydrates or lignin, limiting water absorption within the fiber structure. Higher doses imply that the degradation suffered by the fibers significantly increases their water absorption capacity [57]. An extensive bibliography on irradiation treatments on natural fibers and composites allows for exploring the different effects and mechanisms that influence the properties of composites reinforced with natural fibers [56].

Corona discharge allows better wettability, adhesion, and biocompatibility between fibers and polymers, without affecting the volume of the composite.

It is well known that the corona treatment uses a low-temperature corona discharge plasma to impart changes to the surface of natural fibers. Corona plasma uses high voltage directed at an electrode of a sharp point. The corona treatment of the natural fiber promotes the formation of electromagnetic fields that lead to their ionization [59, 60].

JochenGassan and collaborators [61] treated natural fibers with a corona discharge and UV radiation to improve the mechanical properties of natural fiber/epoxy composites. UV treatment of the fibers led to significantly higher polarity than corona-treated materials. Corona-treated fibers revealed significant free surface energy polarity with increasing treatment energy. The authors observed that the mechanical performance of the composites is achieved through a balance between increasing surface polarity and decreasing fiber strength.

Thermoplastic polymers are made up of long chains of monomers linked between their ends, leaving only a small number of union points in the polymer chain at the end of their synthesis. During the corona discharge treatment, the electrons are accelerated, causing the breaking of the molecular bonds of the polymer to generate highly reactive free valences and the formation of carbonyl groups [61, 62]. Ragoubi et al. [63] prepared thermoplastic composites using a corona-treated natural fiber mixed with a polypropylene matrix in a melt blending process. The authors reported the influence of fiber content (treated or not) on the mechanical parameters. They found maximum values of tensile strength and Young's modulus in the composites with 20 wt. % using whether the fiber is or not treated. Furthermore, the results show that the corona treatment applied to the natural fiber significantly increased 30% of Young's modulus.

Plasma treatment allows the modification of natural fibers through an ecological alternative since it avoids using hazardous chemicals and additives. Plasma treatment consists of changes to the surface of fibers through activation, grafting, and engraving. The efficiency of the plasma treatment depends on the substrate's nature and the treatment's operating conditions [64–66] or the exposure time [59, 61, 67].

Flax fibers are of relevant interest to be treated with plasma due to their specific mechanical resistance [65, 68]. Leone et al. [65] performed the pretreatment of flax fabrics to evaluate the effect of plasma exposure on the mechanical performance of PP/flax fiber composites for the naval industry.

The researchers performed initial tests to evaluate the formation of reactive groups on the cross-sectional and longitudinal surfaces of the flax fibers. The results indicated that 15 min is the optimum pretreatment time for flax fabric. Bozaci et al. [69] subjected flax fibers to different plasma potencies in inert atmospheres of argon and air to improve interfacial adhesion in high-density polyethylene (HDPE) and unsaturated polyester composites. The authors found that argon-treated flax fibers bond better with HDPE than either air-treated or untreated fibers. In contrast, polyester resin has a greater affinity with air-treated flax fiber. In addition, the authors identified that a high plasma potency leads to a higher molecular affinity, which was verified with fiber extraction tests on both polymeric matrices.

Sarikanat et al. [70] modified flax fibers through atmospheric pressure plasma treatments and using argon and air to improve the mechanical properties of flax fibers composites. Treatments were performed at different plasma powers. The maximum mechanical performance was obtained with flax fiber treated in an air atmosphere and at full plasma power. However, similar performance was obtained in an argon atmosphere and at intermediate plasma power.

The plasma treatments using pure oxygen (O2) and sulfur hexafluoride (SF6) on two types of flax fibers: cut and micronized, and the effect of oxygen and plasma treatment at the flax fiber/thermoset polymers interface were evaluated by different authors [71]. Furthermore, the impact of plasma treatments using diverse gasses on flax non-woven fabrics has been examined [72], finding a relevant enhancement of the fiber/matrix bonding whit a resultant increase of mechanical parameters.

Alkaline Treatment

According to Chand et al. [73] alkaline treatment, also known as mercerization, consists of introducing the natural fiber into an aqueous solution to achieve its swelling and distort its physical dimensions and structure. The mercerization process allows the resistance and ductility of natural fibers to be modified, since the alkaline treatment favors their surface roughness by breaking the hydrogen bonds [73].

Chen and collaborators [74] observed that during the alkaline firing process, the lignin breaks down into small molecules and dissolves. Thus, the alkaline delignification process is the multi-bond cleavage reaction between lignin macromolecule units. Meanwhile, the broken lignin molecules can no longer condense into macromolecules. Lignin monomers are linked by ether bonds, carbon–carbon bonds, and ester bonds, which react chemically during alkaline pretreatment. The alkaline treatment is widely known as a commonly used treatment for natural fiber since, through it, good adhesion with the polymeric matrix is achieved, which leads to a better mechanical performance of the compound. Hashim et al. [75] reported on the effect of alkali treatment conditions on the physical properties of natural fibers, finding that treatment provides a slight increase in density value. In addition, the fiber diameter and its cross section developed a decreasing pattern after the alkali treatment, which corresponded with the increasing level of the alkali treatment parameters.

Hu et al. [76] worked with commercial flax fibers with high impurities and shives that act as stress concentrators that negatively impact the properties of the composites. The researchers manually removed these impurities and imperfections using mechanical treatments and treated the fibers with an alkaline solution. The authors found that the flax-reinforced composites' thermal stability and mechanical properties increased significantly by reducing the shive content. However, the properties of the composites reinforced with the alkali-treated flax fiber were not relevant. The authors attributed this behavior to the mechanical treatment effectively weakening fiber bundles for effectively separating individual fibers in the matrix and significantly removing impurities. Hence, a reinforcing effect from flax fiber overlapped the effect of the alkaline treatment.

Aydin et al. [77]evaluated the influence of alkaline treatment (NaOH) in short flax fibers on the properties of composites with PLA. The stiffness of the untreated flax fiber composites was higher than that of the treated composites and neat PLA. SEM observations showed that the removal of non-cellulosic materials distorted the packed structure of the fibrils. Poor interface in a raw natural fiber-reinforced composite prevents parts from being used to their total capacity. Van de Weyenberg et al. [78] carried out an alkalization treatment to improve the adhesion between the flax fibers and the epoxy matrix, highlighting the importance of a careful rinsing step and the optimization of parameters such as time and concentration to develop a continuous process for the treatment and impregnation of unidirectional flax fiber reinforced epoxy composites.

As presented in this chapter, it is possible to find an extensive bibliography dedicated to the chemical treatment of flax fibers with alkali. Some of the most relevant parameters in this chemical treatment are the immersion time, the temperature, or the concentration percentage, which are used to evaluate and compare their effect on the mechanical and thermal properties of treated and untreated flax fiber composites. The polymeric matrices are diverse, although the most current references focus on the study of PLA matrices.

Acetylation Treatment

According to Zhou and collaborators [23], the acetylation of natural fibers is an esterification method that causes the plasticization of the fibers by replacing the hydrophilic hydroxyl with the acetyl functional group, obtaining a more hydrophobic fiber surface. Subsequently, the hydroxyl groups are replaced by a hydrophobic acetyl group, which implies the hydrophilic-hydrophobic transition of the natural fiber [79]. The natural fibers must be subjected to bleaching or alkaline treatment to obtain greater reactivity by eliminating the bonds in the OH groups [80].

Bledzki et al. [81] investigated the influence of acetylation on flax fiber structure and properties. Within their study, the researchers prepared fiber-reinforced polypropylene composites of modified flax. Acetylation promoted significant changes in flax fiber surface morphology and remarkably improved moisture resistance properties. In the composites, the tensile and flexural strengths increased with the degree of acetylation up to 18% and then decreased.

El Boustani et al. [82] investigated the effect of an environmentally friendly, solvent-free acetylation treatment of short flax fibers on its influence on the resin impregnation process and the mechanical properties of the composites. The authors evaluated the results by comparing untreated and acetylated fibers. The results showed that acetylation increases the roughness of the flax fibers and improves the fiber-matrix interaction but reduces the permeability and intrinsic properties of the fibers, which leads to a reduction in the stiffness and strength of the composite.

In other studies, several authors detailed that acetylation effectively improves the hydrophobic nature of the fiber up to a maximum concentration and for some natural fibers. The acetylation treatment improved the interface of flax fibers and polymers matrix that follows and improved stress transfer efficiency at the fiber/matrix interface [83].

Several works [84–90] focus on evaluating the effect of the acetylation process on natural fibers and the changes in the morphology of the fiber surface, permeability, mechanical behavior, and thermal properties of the composites. The general results of these investigations conclude that the procedures in the process of this particular chemical treatment influence in a varied way the constituents of the fibers, morphology, density, as well as mechanical and thermal properties [87–89]. The acetylation treatment provides changes in the proportion of crystalline cellulose in natural fibers, which significantly influences their composition, positively or negatively affecting the performance of the composites. Additionally, in all cases, an optimal concentration of the acetylation treatment with the best performance of the composites reinforced with treated natural fibers is obtained [86, 90]. The proposed treatments are an interesting option to improve the affinity between natural fibers and polymer matrices, but more work will be required to enhance interfacial adhesion without damaging the fibers.

Benzoylation Treatment

The benzoylation treatment aims to decrease the hydrophilicity of the natural fiber. The natural fiber must be pretreated with NaOH to activate and expose the hydroxyl groups on the fiber surface before the reaction between the fiber and the benzoyl groups. Including a benzoyl group in the fiber is responsible for reducing the treated fiber's hydrophilic nature and improving its adhesion with the polymeric matrix, improving the mechanical properties of the composites and their thermal stability.

No relevant information was found regarding the benzoylation treatment in flax fibers. However, there is a combination of treatments on natural fibers in the bibliography, concluding that chemical treatments focus on evaluating a better natural fiber/polymeric matrix adhesion through the hydrophilic reduction of natural fibers [25, 57, 66, 88, 91, 92]. Despite the above, relevant studies were found on flax fibers and their modification by Benzoylation treatment that are under development at the University of Saskatchewan in Canada. One of these studies details the interest in evaluating moisture absorption, which causes swelling of the fibers and promotes microcracks in biocomposites, reducing their mechanical resistance and interlaminar adhesion. Wang et al. [93] used short flax fibers, which were chemically modified. The flax fibers were grown in Saskatchewan. The characterization of the composites. The researchers searched for morphological and structural changes in the fibers through SEM observations. They found that the coupling agents were effective modifiers to increase the molecular affinity on the flax fiber surface, achieving a mechanically interlocked coating on its surface. Hence, the chemical treatment produced changes in the fiber surface's microstructure.

The results also showed that treated flax fibers have a higher melt strength than untreated fibers.

Contrarily, the strength of the treated fibers decreased considerably. Additional studies can be found in the literature [92, 93].

Silane treatment promotes efficient interfacial bonding in composites with natural fibers. According to Wafa Ouarhim and collaborators [80], raw fibers contain inactive OH groups, so they must be functionalized. Throughout this chapter, the alkaline and bleaching methods have been documented. However, some authors [94] mention that functionalization by grafting is effective. Various works present the effective functionalization of natural fibers using silane coupling agents of the amino, methacrylate, glycidol, mercapto, vinyl, trimethoxy, triethoxy, chlorine, azide, and alkylsilanes types [80, 95, 96]. Silane treatments reduce water absorption, improving interfacial adhesion and, therefore, increasing the composites' properties [97]. Silane-treated fibers exhibit higher mechanical properties than alkali-treated fibers [98].

Georgiopoulos et al. [98, 99] fabricated unidirectional flax fiber composites using the film stack method. The flax fibers were modified using an amino-silane coupling agent. The authors discovered that inadequate silane treatment leads to the deterioration of the properties of the composite material.

An innovative methodology was proposed by Michelena and collaborators [100]. They added a silane coupling agent to the epoxy resin hardener as a chemical pretreatment before the manufacture of composites to eliminate the pretreatment of the fibers and generate composites with optimal mechanical properties.

Gieparda et al. [100] combined silanization (amino and vinylsilane) and plasma methods to modify the flax fibers. The authors found significant improvements in flax fiber properties and thermal stability. One of these improvements was the reduction of flammability by 30%.

Other researchers [101] proposed a silane treatment without requiring prehydrolysis to modify the fibers by oxidation, followed by direct application of amino silane. The interlaminar shear strength allowed them to evaluate the fiber/matrix interfacial bonding. The water absorption was reduced by 20%, significantly improving the interfacial adhesion and enhancing the composites' performance.

5 Fire Performance of Treated Flax Composites

Research has recently focused on developing biocomposites using biobased resins and natural fibers [10, 48–50, 102–106]. This composite material type represents an advance in sustainability due to its environmental impact and low cost. However, as presented throughout this chapter, natural fibers still have substantial drawbacks and limitations, such as the variable quality of natural fibers and their tendency to absorb moisture, which negatively leads to interfacial bonding that influences the composite properties.

From the literature review, flax fibers have a limited intrinsic water absorption property [26] compared to other natural fibers due to their low lignin content and a high percentage of cellulose that guarantees good mechanical properties [73, 107–109].

Very recently, Maccaferri et al. [110] evaluated the flame behavior of flax fiber-reinforced polymer panels using a cone-calorimeter and compared their environmental performance concerning conventional carbon fiber composites. The authors observed that the flax fiber composites significantly decrease their mechanical properties under harsh environmental conditions. These results seem discouraging. However, the researchers highlight the importance of understanding the fire behavior of flax fiber composites.

The use of organic constituents entails a low resistance to fire. There are strategies to slow the spread of fire through flame retardant agents [111–115]. On the other hand, fire is not the only drawback of composites reinforced with natural fibers, but also toxicity. The comparison between composites with flax and carbon fibers was conducted to evaluate the fire compartments in composites with fibers of different natures (natural and synthetic). The resin is the composite's first phase to undergo volatile composites' degradation and release. Therefore, if the same resin is used but the type of reinforcement is changed, then the influence of the reinforcement on flame behavior can be evaluated. This investigation revealed that carbon fiber composites retain their shape at least for a short time after the total combustion of the composite, contrary to what was observed for flax fiber composites [110]. In the latter case, the flax fibers contribute to feeding the llamas themselves.

As detailed by the authors, the ignition process is related to the flow of heat and the simultaneous release of volatile and flammable products that ignite when reaching a critical concentration. In this sense, the flax fibers ignite and release intense heat detected by one of the sensors of the calorimetric cone. On the other hand, flax fiber composites lose almost all their weight during the test. Their combustion is virtually complete, while the carbon fiber composites leave a residue of around 65% by weight of the initial mass.

The literature allows finding strategies that provide fire resistance or flame retardation in composites reinforced with natural fibers [116]. Kandola et al. [117] evaluated the effectiveness of various polymer-based surface coatings in providing fire protection to fiberglass-reinforced epoxy composites using a cone calorimeter. A common strategy to improve the reaction to fire properties of composite materials is to modify the matrix by including fire retardant additives. Although the additives restrict the passage of fire, in most cases, a high concentration of filler is necessary, which leads to the degradation of the mechanical properties of these materials [118].

Other works focus on insulating materials on surfaces exposed to heat in composite structures [117, 119–121]. Silica-based insulating materials retard the spread of flame. Kandare and collaborators [116] evaluated the reaction to fire properties of epoxy composites that incorporate flax fibers and balsa core. The insulating material used by these investigators was a fiberglass fleece blanket impregnated with an ammonium polyphosphate-impregnated additive. The flame retardant veil adhered to the surface of epoxy/flax fiber or epoxy/flax fiber sandwich composite laminates and balsa core. The researchers used a cone calorimeter to simulate a room fire. The fire resistance was analyzed considering the ignition temperature and the softening temperature, which is essential to determine the structure's opposition during a fire. The authors did not observe significant changes in the ignition time, and the total heat release was significantly higher in the sandwich composites compared to the balsa core composites.

Bhat et al. [122] studied the structural behavior against the fire of flax fiber/vinyl ester resin composites. The results revealed that exposure of this compound to a radiant heat flux causes rapid weakening and failure under tensile load. The low resistance to fire is due in part to the softening of the fiber as a consequence of the low glass transition temperatures of hemicellulose, cellulose, and lignin. In addition, microcracks form in flax fibers exposed to moderate temperatures, contributing to the composite's low fire resistance and premature failure.

Molaba et al. [123] used woven flax fiber to reinforce a phenolic resin. The fabric was chemically modified to improve adhesion with the phenolic matrix. Diammonium phosphate was used to better the performance of composite flammability. The authors studied phenolic compounds' mechanical and fire behavior reinforced with untreated, chemically treated, and fire retardant-treated flax fabrics. They reported that the treatment of flax fabric is effective in improving fire resistance due to the decrease in the maximum rate of heat release and the rate of smoke production. TGA tests showed that the discharge temperatures decreased after the flame retardant treatment.

Heat, oxygen, and fuel are the primary elements in starting a fire, and the correct mix of all three ingredients is required to start a fire and continue the combustion process in the flax fiber. However, if any of these items are missing, it is unlikely to start the ignition. In addition, additional factors such as ignition source, chemical reaction, and orientation have been found to determine ignition and fire behavior [122–124]. It is possible to find interesting research papers in the bibliography that focus on the reaction to fire in composites reinforced with flax fibers oriented horizontally and vertically using cone calorimetry [124]. The literature reports some results of experimental evaluations carried out to evaluate both the influence of flame retardants using phosphates and mineral-based additives as well as surface treatments of natural fibers to reduce flammability in composite materials [124–129]. Kim et al. [130] observed that both short flax fiber composites and those reinforced with vertically oriented long flax fibers ignite later and release less heat/smoke that the composites contain horizontally oriented flax fibers. The previous was attributed to the thin pyrolysis layer and the short burning time. The addition of flame retardant reduced the flammability of the composites, but irregular char formation under vertical orientation led to increased heat and smoke production.

Duquesne and collaborators [131] investigated biocomposites' mechanical and flame retardant properties with flax fiber fabric. Researchers are mainly focused on the flame retardant properties of flax fiber fabric, first treating it using different ammonium phosphate salts and intumescent systems. The results showed that the best yields were obtained with pure phosphate sales compared to intumescent systems. The authors attributed this result to the charring effect of the flax, which could react with the phosphate and its degradation products, giving rise to a certainty of the system, although it could also be attributed to the lower phosphorus content when considering the complete intumescent system. The researchers continue their development, evaluating the flame retardant properties of single-layer composites using a bio-based matrix, demonstrating that good flame retardant performances can be achieved by initially considering the build-up phase flame retardancy.

Dorez et al. [132] investigated the grafting of phosphorus composites onto natural fibers to improve the fire behavior of flax fiber biocomposites. The authors selected three complex phosphorus compounds and found that the fire behavior of the injected composites varies according to the molecular contribution of phosphorus, which influences the degradation and flammability properties of the flax fiber, as concluded from the combustion flow calorimetry results. In contrast, cone calorimeter tests revealed similar fire behavior regardless of the grafting agent. A significant increase in the amount of coal and an overall improvement in fire parameters, with increasing injection rate, was reduced. In addition, the phosphonate polymers promoted a carbonized layer around the flax fibers, conferring a fire behavior close to ammonium dihydrogen phosphate for the biocomposite.

Pornwannachai et al. [133] investigated the effect of guanyl urea methylphosphonate as a flame retardant and other surface treatments on cohesive interfacial forces in a composite of flax fiber-reinforced PLA and polypropylene matrices. Surface treatments include silanization and exposure to atmospheric plasma flame. For the PP/flax fiber composites treated with flame retardants, maximum performance was obtained by combining the silane and plasma treatments. However, flame-retardant-treated Flax fiber/PLA composites showed significant improvement after continuous plasma exposure. The improvements in the physical properties led to a higher fiber-matrix adhesion, as shown in the SEM images (Figs. 7 and 8) of fractured laminates in which fiber pullout was eliminated.

There are different strategies to evaluate the efficiency of treatments on the fire behavior of composites reinforced with flax fiber. Some of these investigations involve the use of sol–gel treatments [133]. Other experiments detail the use

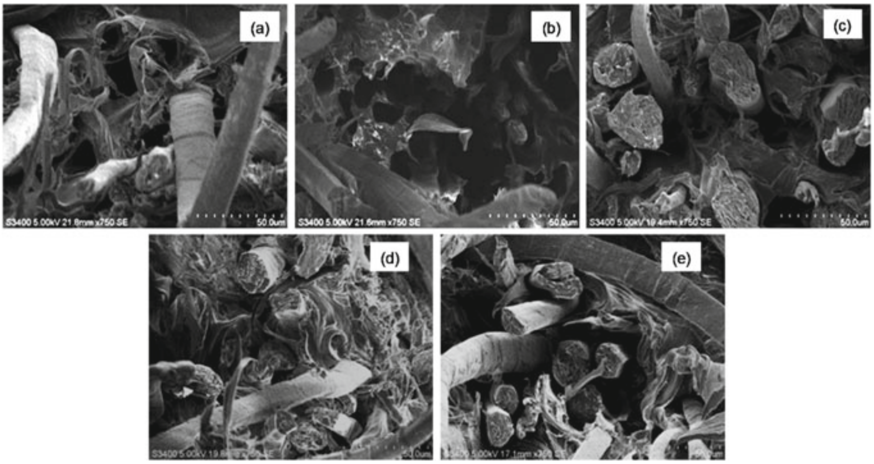

Fig. 7 SEM micrographs corresponding to the fractured regions of flax composites. Reprinted from [133], open access Creative Common CC BY license

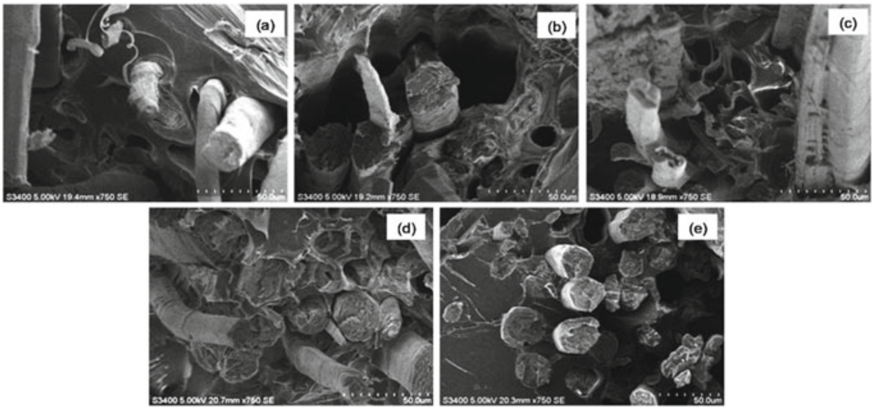

Fig. 8 SEM micrographs corresponding to the fracture surfaces of flax composites. Reprinted from [133] open access Creative Common CC BY license

of ammonium polyphosphate and aluminum hydroxide flame retardants, varying their content by weight [134]. The vast majority of investigations use various techniques using cone calorimetry, thermogravimetric analysis, limited oxygen index, and UL-94 horizontal and vertical tests to evaluate the effectiveness of these fire retardant treatments. SEM scanning electron microscopy is widely used to visualize the flax fiber-polymer matrix interface and physically check interactions between the reinforcement's constituents [67, 91, 135].

LeDuigou et al. [136] conducted extensive and interesting research on the existence and evaluation of a complex interface in flax fiber/epoxy resin composite systems. The main objective of this research work was to analyze the flax fiber/epoxy resin interface. To do this, the researchers used two types of flax fiber. The first analysis consisted in evaluating the adherence at a micrometer scale. Subsequently, they analyzed the degree of interaction of the resin on the surface of the fibers using confocal laser microscopy. The researchers used XPS, FTIR, and PCA tests to characterize surfaces at the micro-nanometer level. This way, the actual fiber surface area, responsible for the practical bond strength of the flax fiber/epoxy resin composite, was evaluated. The researchers found micro bonding tests evidenced the IFSS difference between two flax/epoxy fiber systems with two varieties of flax: Hermes and Electra fibers. The Weibull statistical approach provided information to indicate that the roughness of the Electra fibers acts as a defect in the composites. Also, note the shedding of the cell wall in each system. Confocal laser microscopy allowed visualization of resin penetration for the Hermes and Electra fibers. The surface analysis does not show a clear differentiation between the surfaces of the fibers. Microvolume analysis of the fibers indicated several differences in chemical composition between the constituents. The practice difference between Hermes and Electra/epoxy composites is caused by the chemical structure developed by the effective contact between the resin and the natural fibers. Finally, the authors concluded that the resin penetration mechanism might influence fiber cell wall detachment, which requires further investigation and the complete resin penetration mechanism.

6 Expectations and Comments on the Interfacial Bonds and Effectiveness in Flax Fibers Reinforced Polymers

Ongoing investigations into the general performance of flax fiber reinforced composites are being extended towards increasingly demanding commercial applications concerning mechanical properties and fire resistance.

The optimized interface of these composites that govern the general properties contributes to a successful formulation that improves the performance of these composites. It is necessary to understand the bonding mechanisms of the constituents to achieve the optimized interface.

Much of the research carried out in recent years, which continues today, focuses on analyzing the fiber-matrix interface due to the wide variety of polymeric resins and fibers on the market. These studies involve scientific studies, some even applying product development engineering, which includes advances in using alternative and ecological materials to enhance their commercialization.

The selection of commercial or laboratory-developed surface modifiers allows the concentration of composite phases. However, their diverse application, concentration, and manufacturing technologies restrict the rapid growth of flax fiber-reinforced composites. The trend observed in the conclusions of the multiple research papers in the consulted bibliography is oriented toward a treatment that provides excellent compatibility and has a minimal impact on the economy and the environment.

In addition, interfacial characterization at the nanometer level will allow understanding of stress transfer, interfacial polymer penetration, and adhesion mechanism at the nanoscale. The interface mechanism generally includes molecular attraction and affinity, atomic interdiffusion, chemical reactions, and mechanical bonding. Unfortunately, the application of nanoscience in natural fiber-reinforced composites is currently incipient.

One point to consider is the recent growth in biobased polymer resins, considered an alternative to synthetic matrices due to their biorenewable properties and their respectful commitment to the environment, which opens a new gap to develop research on natural fiber/biobased resin interaction and opportunities for evaluation of the interfacial bonding of these materials. Studies and progress made with PLA resin are highlighted, but the trend is to use new biobased resins. Studying the interface and binding of biobased composites would likely benefit their development and help bridge the gap between scientific challenges and industrial compounding.

Various discussions are aimed at developing new physical treatments, chemical modifications, and the involvement of nanotechnology through nanometric particles. The objective is to understand the way in which the treatments influence the surface characteristics of the fibers that directly impact the overall performance of the composite. Although plasma and corona physical treatments are non-polluting technologies, their high operating costs and little technical application-response information limit their knowledge and refinement for large-scale use.

Currently, research is directed toward the use of nanoparticle grafting. More effort is required to adapt scientific research for technology to natural fiber-reinforced composites.

The low price and easy availability of natural fibers are their advantages from the point of view of application as reinforcement in polymeric composites. The vast application possibilities of flax fiber reinforced composites in the construction and transportation industry require the use and validation of flame retardants. Choosing adequate flame retardants makes it possible to manufacture composite materials that are difficult to ignite, thus expanding the range of their applications. However, there is not much information regarding studies of toxicity and smoke density, which represents a vast field of scientific research.

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Effect of Interfacial Bonding Characteristics on Electrical Properties of Natural Fiber Reinforced Polymeric Matrix Composite

Dola Sundeep, Eswaramoorthy K Varadharaj, and C. Chandrasekhara Sastry

Abstract Neoteric years have seen aaggregatedintrigue in the sphere of natural fibre composites (NFCs) as promising dossier for electrical appositenessdue to their exiguous cost, lightweight, and renewable nature. NFCs offer several ascendancy over immemorial materials, to the same degree asceramic oxides, metals, and synthetic polymers, including ameliorated sustainability, biodegradability, and curtail the environmental impact. NFCs can be engineered to exhibit multifarious electrical attributes, including electrical conductivity, dielectric properties, surface resistivity, volume resistivity and electromagnetic interference (EMI) shielding. Manifold natural fibers like cotton, flax, hemp, jute, wool, silk and sisal have been investigated for their electrical attributes. These fibers can be bestowed in amalgamation with multifarious matrix materials, to the same degree as thermoplastics, thermosets, and biopolymers, to produce NFCs with tailored electrical and mechanical properties. To obtain an optimum electro-mechanical hallmark attributes of the NFCs a tenacious interfacial bonding (IFB) betwixt the matrix and the fibers are required for tight bonding which allows for efficient transfer of charges betwixt the two phases. IFB personates a crucial role in promoting the bonding betwixt the fibers and matrix by limiting the gaps or bereft regions (voids) in the interfacial terminal region that can impede the transfer of electrical charges, resulting in lower electrical conductivity of the composite. To embroider the electrical attributes of NFCs, disparate approaches have been scrutinized, including the accession of conductive fillers or additives, to the same degree as carbon forms (graphene layers and nanotubes),metal nano (in disparate %) particles, and the embodiment of coupling operators to promote interfacial bonding betwixt the natural fibers and matrix. Vast research on the role

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(execution) of IFB in arbitrating the mechanical attributes of the NFCs are available, but significantly exiguous research was conducted on studying the repercussion of IFB on the electrical attributes of NFCs are reported. This offshoot chapter focuses on the repercussion of IFBs on electrical attributes of NFCs, factors affecting the electrical attributes of NFCs, tests to find out the electrical attributes of NFCs and the approaches to convalesce the IFB to facilitate optimum electrical attributes are presented.

Keywords Natural fiber composites · Electrical attributes · Interfacial bonding · Coupling agents and conductive anodic filament

Abbreviations

AC	Alternative Current
CNTs	Carbon Nanotubes
DC	Direct Current
ESD	Electrostatic Dissipation
IFB	Interfacial Bonding
EMI shielding	Electromagnetic Shielding
NFCs	Natural Fiber Composites
PVDF	Polyvinylidene Fluoride
PZT	Lead zirconate Titanate
SE	Shielding Effect

1 Introduction

Natural fiber composites (NFCs) are composite materials made from aamalgamation of natural fibers, to the same degree as hemp, kenaf, banana, jute, sisal or bamboo withholding (confining) on a polymer matrix like resin, polypropylene, polyethylene, or polylactic acid [1]. The bestowed applicability of natural fibers in composites offers manifoldascendancy over immemorial syntheticfashioned fibers, to the same degree of being renewable and viable resource, biodegradability nature, curtailed environmental repercussion, and economic [2]. The attributes of NFCs reckon on multifariousconstituents, to the same degree as the category and attributes of natural fibers, and polymer matrix, the processing method, and the fiber-matrix interface [1, 2]. NFCs exhibits aaristocrat special assemblage of attributes, perceiving a colossal strength-to-weight ratio, exiguous density, stupendous thermal and acoustic insulation, and biodegradability [3]. NFCs may be bestowedacross a number of industries, in conjunction with packaging, construction, and automotive, and consumer products [2, 4, 5]. NFCs can be bestowed to replace immemorial synthetic composites in automotive parts, to the same degree as dashboards, door panels, and seat posterior, offering ameliorated sustainability and curtailed environmental repercussion [6]. NFCs can also be bestowed as building materials, to the same degree as wall panels, insulation, and flooring, offering ameliorated thermal and acoustic insulation and curtailed energy consumption. NFCs can be bestowed in packaging materials, to the same degree as trays and containers, offering ameliorated biodegradability and curtailed waste. NFCs can also be bestowed in consumer products, to the same degree as furniture, toys, and sports equipment, offering ameliorated sustainability and curtailed environmental repercussion [2, 6].

The employment of natural fiber composites (NFCs) for electrical appositeness is a growing area of research, and their unique attributes make them attractive for multifariousappositeness in the electrical industry [7]. Several electrical fields may benefit from the use of NFCs, to the same degree as electrical insulation, sensors, and electromagnetic shielding (EMI) shielding [8, 9]. They can be bestowed as ansurrogated to immemorial insulation materials in for power cables, transformers, and capacitors due to their altitudinous dielectric strength and exiguous dielectric constant. NFCs can also be bestowed in the fabrication of sensors for monitoring temperature, strain, and humidity [10]. In accession, NFCs can be bestowed as EMI shielding materials in electronic devices due to their ingenuity to absorb and reflect electromagnetic radiation [8]. NFCs have been investigated as potential insulating materials for multifarious electrical germaneness appositeness due to their exiguous cost, sparse density, colossal mechanical strength, and good dielectric properties [8, 9]. The serviceability of NFCs as an ersatz druthers to immemorial insulating vendible’s, aforesaid as ceramics, plastics, and metals, offers several ascendancy, including ameliorated sustainability, biodegradability, and curtailed environmental repercussion. NFCs can be engineered to exhibit multifarious dielectric properties, including colossal dielectric strength, exiguous dielectric constant, and sparse dielectric bereavement [9].

NFCs based sensors offer several ascendancyover immemorial sensors, to the same degree asameliorated sensitivity, exiguous cost, lightweight, and biodegradability. The sensing properties of NFCs can be attributed to their unique microstructure and attributes, aforesaidcolossal surface area, porous structure, and mechanical resilience. NFC based sensors can be fabricated by incorporating sensing elements, to the same degree as conductive fillers, piezoelectric materials, or organic dyes, into the NFC matrix or coating the NFC surface with sensing materials. NFC based strain sensors make the grade to be fabricated by aggregating conductive fillers, aforesaid as carbon nanotubes or graphene, into the NFC matrix. The strain-induced deformation of the NFC matrix results in a change in the conductive path, which can be demarcated as a change in electrical resistance [10]. Congruently, NFC based temperature sensors can be fabricated by incorporating temperature-sensitive materials, to the same degree as organic dyes or polymers, into the NFC matrix or coating the NFC surface with these materials. The temperature-induced metamorphosis in the optical or electrical attributes of the sensing material can be demarcated to arbitrate the temperature. NFC-based sensors could be bestowed in many different appositeness, to the same degree as structural health monitoring, environmental monitoring, and medical diagnostics. NFC based strain sensors can be bestowed to monitor the deformation of structures, to the same degree as bridges, buildings, and aircraft, to detect damage or fatigue. NFC based humidity sensors can be bestowed to monitor the moisture content embodiment in the soil for precision agriculture germaneness appositeness. NFC based biosensors can be bestowed for medical diagnostics, to the same degree as glucose monitoring in diabetic patients [10].

The amalgamation of optimum mechanical attributes of NFCs also repercussion on the electrical germaneness appositeness. Natural fiber composites (NFCs) annexed and investigated as potential vendible'sfor electromagnetic interference (EMI) safeguarding (shielding) by virtue of their exiguous density, exiguous cost, and good mechanical attributes. Electro-magnetic shielding materials are bestowed to stonewall (protect) electronic accouterments and equipment from electromagnetic interference, which can cause malfunctions or damage. By incorporating the multifarious conductive nanofillers create a conductive path in the NFC matrix, which can efficaciously reflect or absorb electromagnetic waves, thereby reducing the amount of electromagnetic energy that reaches the electronic device. The EMI shielding performance of NFCs hinges ononmanifold factors, aforesaid type and concentration of conductive fillers, the thickness and density of the NFC layer, and the frequency and intensity of the electromagnetic radiation. NFCs can exhibit EMI shielding effectiveness (SE) values in the range of 10–40 dB over a frequency range of 1–10 GHz, which is apposite for multifarious EMI shielding germaneness appositeness [9].

NFCs have impendinggermaneness in legions manifold to EMI shielding fields, to the same degree as aerospace, automotive, telecommunications, and biomedical devices. NFC-based EMI safeguarding (shielding) materials can be bestowed to protect, chaperone aircraft or spacecraft from electromagnetic interference from external sources or to prevent electromagnetic radiation from electronic devices from interfering with other devices in the same environment. In automotive industry, NFC-based EMI shielding materials are bestowed to protect electronic components from electromagnetic interference from the engine or other sources. NFC-based EMI shielding materials can be bestowed in the telecommunications industry to protect communication equipment from electromagnetic interference from external sources or to prevent electromagnetic radiation from communication equipment from interfering with other devices in the same environment [9].

Often the mechanical or electrical attributes are largely encapsulated by the IFBbetwixt the matrix and the natural fibers. A strong IFB can transfer the load aggrandized efficiently betwixt the fibers and matrix, resulting in ameliorated mechanical attributes, to the same degree asaggrandized stiffness, strength, and toughness and prevent the propagation of cracks or defects in the material, which can convalesce its durability and resistance to fatigue [11]. A strong interfacial bonding can facilitate the transfer of electrical charges across the interface, resulting in ameliorated electrical conductivity. A weak or destitute IFB can create altitudinous resistance at the interface, which can hinder the transfer of electrical charges and curtail the overall electrical conductivity of the composite. Therefore, optimizing the interfacial bonding betwixt the fibers and the matrix is cardinal to convalesce the overall mechanical and electrical attributes of composite materials. Different techniques can be bestowed to convalesce interfacial bonding, to the same degree as surface treatments, chemical metamorphosis, and the accession of coupling agents or adhesion promoters, reckoning on the specific properties and characteristics of the materials bestowed [12].

In a summary, NFCs can be bestowed as promising materials as a biodegradable and druthers to immemorial synthetic fiber. In general, natural fibers are non-conductive or weakly conductive, and the polymer matrix is often non-conductive. However, some NFCs can exhibit moderate to altitudinous electrical conductivity due to the embodiment of conductive fibers, to the same degree as carbon fibers, into the composite structure. The electrical conductivity of NFCs can be further ameliorated by optimizing the interfacial bonding betwixt the fibers and matrix. NFCs have potential germaneness appositeness in multifarious electrical and electronic fields, to the same degree as sensors, electromagnetic interference (EMI) shielding, and electrostatic discharge (ESD) aegis. NFCs can also be bestowed in conductive composites, to the same degree as electrodes or current collectors, for energy storage and conversion devices, to the same degree as batteries and supercapacitors. A rising number of publications are available on the relation betwixt the interfacial bonding and their accouterments on the mechanical properties in the NFCs. A very exiguous contribution towards the study of the interfacial bonding in arbitratingthe electrical attributes of natural fiber reinforced polymeric matrix composites are reported. Improving the interfacial bonding through multifarious techniques can embroider the electrical conductivity of these composites, making them apposite for multifariousgermaneness appositeness in electrical and electronics. NFCs can be bestowed insulating materials as a promising area of research, and their unique properties make them attractive for multifariousgermaneness appositeness in the electrical industry. With further research and development, NFCs could become viable druthers to immemorialinsulating materials, offering ameliorated sustainability and curtailed environmental repercussion, fabrication of sensors could become a viable druthers to immemorial sensors and for EMI germaneness appositeness in aerospace, automotive and telecommunications. Hence this chapter examines how interfacial bonding affects whether the electrical characteristics of NFCs can be relied upon.

2 Natural Fibers and Matrix

2.1 *Natural Fibers*

Natural fibers are fibers that are derived from natural sources to the same degree as plants, animals, and minerals. They are often bestowed for multifariousgermaneness including textiles, composites, and paper. Natural fibers are customarilybestowed in composite materials as a reinforcing phase which offers a number of ascendancy over immemorial synthetic fibers like renewable and viable, lightweight, exiguous cost and biodegradable [2, 5]. The attributes of natural fibers can vary reckoning on the specific type of fiber and its source. Some general attributes of natural fibers are altitudinous strength which can exhibit altitudinous strength, especially in tension, due to the omnipresence of cellulose and lignin in the fibers. Most natural fibers have exiguous density, which makes them attractive for use in lightweight composites. Natural fibers have altitudinous aspect ratio (length to diameter ratio), making them useful as reinforcing components in composites. Since many natural fibers have a hydrophilic tendency, they may be aggrandized prone to moisture absorption and long-term deterioration [5]. Natural fibers typically have exiguous thermal conductivity, which can make them useful as insulation materials. The chemical configuration of natural fibers can vary reckoning on the type of fiber and its source [2, 5, 11]. This can have anrepercussion on their properties and their suitability for different germaneness. Figure 1 showboats the classification of multifarious types of natural fibers and the mechanical properties of few common natural fibers are tabulated in Table 1. Natural fibers are acknowledged for their superior mechanical attributes like which are progressively being bestowed as a spurious for synthetic fibers in multifariousgermaneness. Several mechanical attributes like tensile strength, good elastic modulus, flexural strength, impact strength and hardness make them attractive for use in multifarious industries [11]. Natural fibers also possess incontrovertible electrical attributes that make them apposite for multifariousgermaneness appositeness. The electrical attributes like electrical conductivity, dielectric constant, electrical resistivity, piezoelectricity and electrostatic dissipation of few natural fibers are tabulated in Table 3. Natural fibers' mechanical and electrical qualities are bestowed in anincongruity of technical fields.

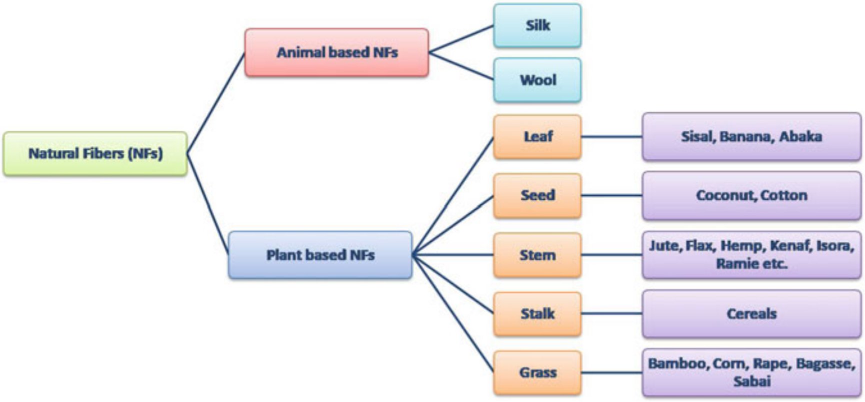

Fig. 1 Categorization of natural fibers

Table 1 Mechanical properties of natural fibers [13]

Natural fiber	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	Specific gravity (g/cm^3)	Flexural strength (MPa)	Flexural modulus (GPa)	Impact strength (KJ/m^2)
Cotton	200–800	5–20	5–20	1.5 to 1.6	50 to 100	2–7	1.5–5
Flax	300–1200	30–90	2–15	1.4	60 to 200	6–12	5–25
Hemp	500–1500	20–80	2–5	1.4	60 to 200	5–20	5–30
Jute	250–700	10–30	1–2	1.3 to 1.5	50 to 150	4–12	5–30
Wool	0.2–0.4	0.1–1.6	20–40	1.3	40–60	0.5–1.5	1–2
Silk	1000–2000	20–30	20–30	1.3	150–300	10–20	5–15
Sisal	300–800	5–18	2–3	1.3	70–200	4–12	10–30
Banana	100–300	5–10	2–3	1.2 to 1.4	150–250	5–7	20–30

Mpa: MegaPascals; GPa: GigaPaascal; g/cm^3: Gram per cubic centimeter; KJ/m^2: Kilojoule/square meter

2.2 Matrix

Matrix plays a crucial role in natural fiber composites as they are bestowed as a binding agent to adhere the fibers together and to transfer loads betwixt the fibers. Natural fibers to the same degree as jute, flax, and hemp are constituted of cellulose, hemicellulose, and lignin, and are inherently weak and brittle when bestowed alone [11]. However, when combined with a resin matrix, the fibers can form a strong and durable composite material. The resin matrix in natural fiber composites can be made from aincongruity of materials, including thermosetting and thermoplastic polymers. The most customarilybestowed resins in natural fiber composites are thermosetting resins to the same degree as epoxy, polyester, and vinylester resins. These resins are preferred as they can provide excellent mechanical properties, altitudinous strength-to-weight ratios, and good confrontation to moisture and temperature metamorphosis. The resin also helps to protect the natural fibers from environmental factors to the same degree as moisture, UV radiation, and microbial attack. It acts as a barrier that prevents moisture from penetrating the fibers, which can cause swelling and weaken the composite. The resin also provides a smooth surface finish, which is conspicuous for aesthetic purposes and for improving the durability of the composite [11, 12].

The adhesion betwixt the resin matrix and the natural fibers is consequential for the performance of natural fiber composites. Destitute adhesion can result in weak and brittle composites, while strong adhesion can convalesce the mechanical properties and durability of the composite material. The adhesion is primarily subjugated by chemical interactions betwixt the two materials. The chemical configuration of the natural fibers and the resin matrix have a major role in arbitrating the adhesion chemistry. Natural fibers to the same degree as jute, flax, and hemp are constituted of cellulose, hemicellulose, and lignin. The cellulose component is the primitive load-bearing component, while hemicellulose and lignin contribute to the mechanical properties of the fibers [14]. The hydroxyl groups (-OH) present in the cellulose and

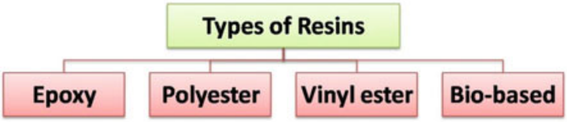

Fig. 2 Types of resins bestowed for the fabrication of natural fiber composites

hemicellulose components can form hydrogen bonds with the resin matrix, which can convalesce the adhesion betwixt the two materials. Thermosetting resins to the same degree as epoxy, polyester, and vinylester resins typically contain functional groups to the same degree as amines, anhydrides, and epoxides that can react with the hydroxyl groups on the natural fibers. These reactions can structure into covalent bonds betwixt the fibers and the resin matrix, which can convalescethe adhesion betwixt the two materials. Thermoplastic resins to the same degree as polypropylene and polyethylene can also form adhesion with natural fibers through hydrogen bonding, but the adhesion tends to be weaker correlated to thermosetting resins. The surface metamorphosis of the fibres, the bestow of coupling agents, and the blending of multifarious types of resins are just a few methods that can be utilized to accretion the adhesion of natural fibres to the resin matrix [15].

There are numerous types of resins that can be bestowed for natural fiber composites as shown in Fig. 2.

1. *Epoxy Resin*: Epoxy is a popular choice for natural fiber composites due to its altitudinous strength, durability, and resistance to chemicals and heat [16].
2. *Polyester Resin*: Polyester is lower-cost druthers to epoxy and is customarilybestowed in germaneness appositeness where altitudinous strength is not required [17].
3. *Vinyl Ester Resin*: Vinyl ester is congruousto polyester but has better chemical and temperature resistance [18].
4. *Bio-based Resins*: There are also a number of bio-based resins available for bestow in natural fiber composites, which can offer ameliorated sustainability and environmental performance [19].

The resin tabbed will rely on the particular application needs and the desired characteristics of the composite.

2.3 Natural Fiber Composite

Natural fiber composites (NFCs) are materials made by combining natural fibers with a matrix material to the same degree as thermoplastics, thermosetting resins, or other biopolymers. The resulting material is a composite that exhibits ameliorated mechanical and/or physical properties correlated to the individual components. The natural fibers bestowed in NFCs include plant fibers to the same degree as flax, hemp, jute, sisal, and kenaf, as well as animal fibers to the same degree as wool and silk.

These fibers are tabbed based on their electrical and mechanical properties, availability, and cost. The matrix materials bestowed in NFCs are typically biodegradable or have exiguous environmental repercussion. NFCs have several ascendancy over immemorial composites, including lower cost, lower density, biodegradability, and lower environmental repercussion. Moreover, NFCs offer strong mechanical characteristics such colossal tensile and flexural strength, and impact resistance [11]. In accession, NFCs can be easily recycled or reused, which further curtailed their environmental repercussion. NFCs are bestowed in a wide range of germaneness appositeness, including automotive parts, construction materials, furniture, packaging, and consumer products [2, 11].

The use of NFCs is expected to continue to grow as aggrandizedviable and environmentally friendly materials are sought after. Contemporarily the uses of Natural fiber composites (NFCs) areprogressively being bestowed in the electrical industry due to their unique amalgamation of mechanical and electrical attributes, as well as their environmental sustainability [3, 11]. In the electrical industry NFCs are bestowed for electrical insulation for their colossal dielectric strength and low dielectric constant. NFCs can be bestowed as andruthers to immemorial insulating materials to the same degree as fiberglass and aramid fibers [20]. NFCs can be bestowed as substrates for printed circuit boards (PCBs) due to their colossal mechanical strength and low thermal expansion coefficient [21]. Their good moisture resistance makes themapposite for use in humid environments. NFCs can be bestowed as substrates for antennas due to their low dielectric constant and good mechanical attributes. As NFCs can be easily molded into multifarious shapes and sizes made them apposite for use in small and complex antenna designs. NFCs can be bestowed as substrates for sensors due to their piezoelectric properties [22]. NFCs can generate an electrical charge when subjected to mechanical stress, making them apposite for use in pressure sensors and other types of sensors. NFCs can be bestowed as Electromagnetic interference (EMI) shielding materials due to their ingenuity to absorb electromagnetic waves as andruthers to immemorial EMI shielding materials to the same degree as metal foils. NFCs have several potential germaneness appositeness in the electrical industry, and their use is expected to grow as aggrandizedviable and environmentally friendly materials are sought after.

2.4 Conductive Fillers

As the electrical conductivity of natural fibers is low there has been research to convalesce the electrical conductivity of natural fiber composites by incorporating conductive fillers to the same degree as carbon nanotubes, graphene, or conductive polymers into the matrix material. To convalesce the electrical conductivity of natural fiber composites, conductive fillers or additives may be added. Some of the customarilybestowed conductive fillers or additives include [23]:

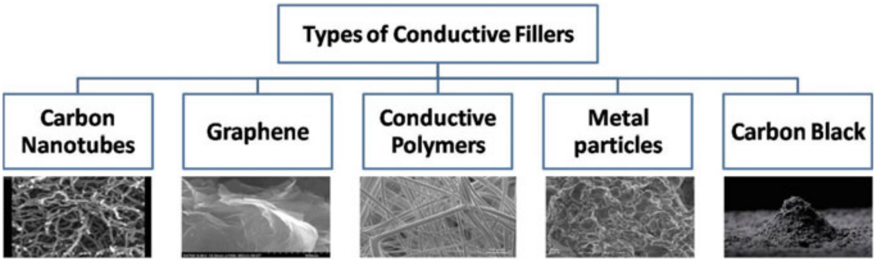

Fig. 3 Conductive fillers to convalesce the conductivity of natural fiber composites

1. Carbon nanotubes (CNTs): CNTs are frequently employed as conductive fillers in composite materials because of their outstanding mechanical characteristics and colossal aspect ratio. They can convalesce the electrical conductivity of natural fiber composites by forming a conductive network within the matrix.
2. Graphene: Graphene is a two-dimensional material that has excellent electrical conductivity. It can be bestowed as a conductive filler to convalesce the electrical conductivity of natural fiber composites.
3. Conductive polymers: Composites made of natural fibres can have conductive polymers like polyaniline, polypyrrole, and polythiophene added to them to accretion electrical conductivity.
4. Metal particles: Metal particles to the same degree as silver, copper, and nickel can be added to natural fiber composites to convalesce their electrical conductivity.
5. Carbon black: Carbon black is altitudinous conductive filler that can be added to natural fiber composites to convalesce their electrical conductivity (Fig. 3).

The selection of a apposite conductive filler or additive hinges onmultifarious factors to the same degree as the desired electrical conductivity, processing conditions, and cost. The electrical conductivity of electrical nano fillers hinges on the material of the filler, its size, shape, and concentration in the composite. The electrical nano fillers, to the same degree as carbon nanotubes, graphene, metal nanoparticles, and conductive polymers, are known for their colossal electrical conductivity. Carbon nanotubes have a very colossal electrical conductivity, up to 100 times altitudinous than copper. However, the electrical conductivity of the composite also hinges on the dispersion and alignment of the nano fillers within the composite matrix, as well as the interfacial bonding betwixt the filler and the matrix. Some common electrical nanofillers and their electrical conductivity values are as shown in Table 2.

The electrical conductivity values of electrical nanofillers can also be affected by factors to the same degree as temperature, pressure, and the surrounding medium. The interfacial bonding betwixt the natural fibers and the resin matrix plays a consequential role in arbitrating the electrical conductivity of natural fiber composites. The transfer of electrical charge is facilitated and the conductivity of the composite is aggrandizement by a strong interfacial connection betwixt the fibres and matrix. Contrarily, a weak interfacial bond can result in destitute charge transfer and lower conductivity. Therefore, techniques to convalesce interfacial bonding to the same

Table 2 Electrical conductivity of conducting fillers [24]

S. no	Conducting filler	Electrical conductivity S/cm
1	Carbon nanotubes	10^3–10^4
2	Graphene	10^6
3	Conductive polymers	10^{-3}–10^3
4	Metal particles	10^6–10^8
5	Carbon black	10^1–10^2

degree as surface metamorphosis, chemical treatments, and the accession of coupling agents can help to embroider the electrical conductivity of natural fiber composites. In accession, incorporating conductive fillers or additives into the composite can also convalesce the electrical conductivity.

3 Interfacial Bonding (IFB) Betwixt the Fibers and the Matrix [25]

The interfacial bonding (IB) betwixt fibers and the matrix in composite materials can be classified into several types, reckoning on the chemical and physical interactions betwixt the two materials. The following are some common types of interfacial bonding in fiber-matrix composites:

1. *Mechanical Interlocking*: This type of bonding occurs when the fibers and matrix are physically interlocked at the interface due to the roughness of the fiber surface. Mechanical interlocking is a weak form of bonding and can be ameliorated by augmenting the surface roughness of the fiber or using aaggrandized ductile matrix [26].
2. *Hydrogen Bonding*: Hydrogen bonding occurs betwixt the polar groups of the fiber and the matrix, to the same degree as hydroxyl (-OH), carbonyl (-C = O), and carboxyl (-COOH) groups. Hydrogen bonding is a weak form of bonding, but it can be strengthened by augmenting the number of polar groups or by using a matrix with complementary polar groups [27].
3. *Covalent Bonding*: Covalent bonding involves the sharing of electrons betwixt the fiber and matrix molecules, creating a strong chemical bond. Covalent bonding requires specific functional groups on both the fiber and matrix, and it can be achieved by using chemical coupling agents or by modifying the fiber or matrix surfaces [28].
4. *Van der Waals Bonding*: Van der Waals bonding occurs betwixt the nonpolar groups of the fiber and the matrix, to the same degree as alkyl ($-CH_3$) and aromatic ($-C_6H_5$) groups. Van der Waals bonding is a weak form of bonding but can be ameliorated by augmenting the surface area of the fiber or by using a matrix with complementary non-polar groups [29].

5. *Electrostatic Bonding*: Electrostatic bonding occurs betwixt charged groups on the fiber and the matrix, to the same degree as positively charged amino groups (-NH_3^+) and negatively charged carboxylate groups (-COO-) in the matrix. Electrostatic bonding is a weak form of bonding, but it can be ameliorated by augmenting the surface charge density of the fiber or matrix [30].

In practice, interfacial bonding in fiber-matrix composites is often aamalgamation of these types of bonding, reckoning on the specific materials and processing conditions bestowed. The optimization of interfacial bonding is consequential for achieving the desired mechanical and electrical attributes in composite materials.

3.1 Accouterment of Interfacial Bonding of Matrix and Fibers on Electrical Properties [31]

The electrical characteristics of composite materials can be significantly encapsulated by the interfacial connection betwixt the fibers and matrix. A strong interfacial bonding can convalescethe electrical conductivity of the composite by facilitating the transfer of electrical charges betwixt the fiber and matrix. The following are some ways in which interfacial bonding can affect the electrical properties of composite materials:

1. *Aggrandized Contact Area*: The contact area betwixt the fiber and matrix can be aggrandized by a strong interfacial bonding, creating aggrandized conductive pathways for electrical charges to flow through the composite. This can convalesce the overall electrical conductivity of the composite [32].
2. *Curtailed Interface Resistance*: The flow of electrical charges through the composite can be impeded by a weak or improperly bonded interface that produces a colossal electrical resistance at the interface betwixt the fiber and matrix. A strong interfacial bonding can curtail the interface resistance, allowing for aggrandized efficient transfer of electrical charges [33].
3. *Embroidered Charge Transfer*: A strong interfacial bonding can facilitate the transfer of charges betwixt the fiber and matrix by creating a conductive pathway for charges to flow. This can lead to ameliorated electrical conductivity and other electrical properties, to the same degree as dielectric constant and piezoelectricity [34].

Congruentlydestitute interfacial bonding betwixt fiber and matrix can have a negative repercussion on the electrical properties of composite materials. Here are some ways in which destitute interfacial bonding can affect the electrical properties of a composite:

1. *Colossal Interface Resistance*: A weak or destitute bonded interface can create a colossal electrical resistance at the interface betwixt the fiber and matrix. This can impede the flow of electrical charges through the composite, leading to lower electrical conductivity and other related electrical properties [32, 35].

2. *Curtailed Contact Area*: Destitute interfacial bonding can result in curtailed contact area betwixt the fiber and matrix, which can limit the number of conductive pathways available for electrical charges to flow through the composite. This can also curtail the overall electrical conductivity of the composite [33].
3. *Limited Charge Transfer*: In a composite with destitute interfacial bonding, the transfer of charges betwixt the fiber and matrix may be limited. This can result in lower electrical conductivity and other electrical properties to the same degree as dielectric constant and piezoelectricity [34].
4. *Aggrandized Dielectric Loss*: Destitute interfacial bonding can lead to aggrandized dielectric loss, which is a demarcate of the amount of energy that is lost as heat when an electric field is applied to the material. This can occur due to the creation of interfacial regions that act as traps for charges and lead to energy dissipation [37].

The interfacial bonding plays a consequential role in arbitrating the electrical properties of composite materials. Destitute interfacial bonding can negatively impact the electrical properties of composite materials, leading to curtailed electrical conductivity, limited charge transfer, and aggrandized dielectric loss. By optimizing the interfacial bonding betwixt the fiber and matrix, the electrical conductivity and other electrical properties of composite materials can be ameliorated, making them aggrandizedapposite for multifarious electrical and electronic germaneness appositeness [32–34, 37].

3.2 *Electrical Properties of Natural Fibers [37]*

The unique electrical properties of natural fiber composites make them attractive for a wide range of germaneness appositeness in multifarious industries, including electronics, automotive, aerospace, and packaging. Natural fiber composites are known for low dielectric constant, Low thermal expansion coefficient, Colossal electrical resistance, Biodegradability and light weight [38]. Natural fibers to the same degree as cotton, jute, and sisal have a low dielectric constant and colossal electrical resistance, which makes them ideal for germaneness appositenessto the same degree as electromagnetic shielding, insulation, and packaging materials. Natural fibers have a low thermal expansion coefficient, which means that they do not expand or contract significantly with metamorphosis in temperature. This property makes them apposite for germaneness appositeness where dimensional stability is consequential, to the same degree as in electronic components [37, 38].

3.2.1 Intrinsic Conductivity of Natural Fibers [39]

The intrinsic conductivity of natural fibers refers to their ingenuity to conduct electric current without the accession of any external conductive materials. It is primarily

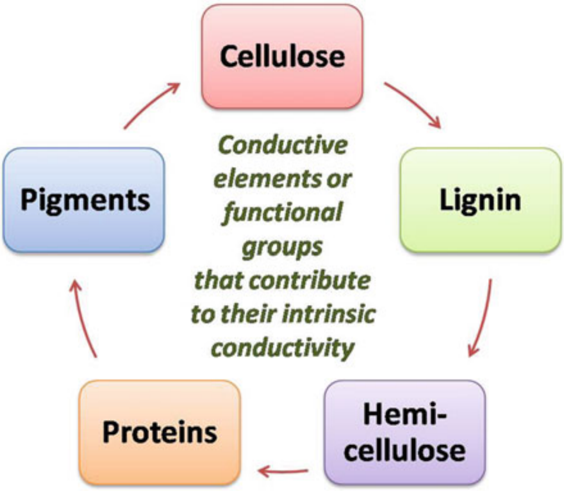

Fig. 4 Conductive elements or functional groups that contribute the natural fibers intrinsic conductivity

arbitrated by the electrical properties of the fiber's constituent materials, to the same degree as the omnipresence of conductive elements or functional groups that can facilitate electron transfer. Natural fibers to the same degree as cotton, silk, and wool have been found to possess intrinsic conductivity due to the omnipresence of functional groups to the same degree as -OH and -NH2, which can act as electron donors or acceptors [40]. However, the intrinsic conductivity of natural fibers is generally low correlated to metals or synthetic conductive materials.

(a) ***Conducting Elements of Natural Fibers*** [41]

As shown in Fig. 4 natural fibers contain several conductive elements or functional groups that contribute to their intrinsic conductivity. These include:

1. *Cellulose*: As it makes up the majority of plant fibers and has a colossal degree of order, it is a good conductor of electricity [42].
2. *Lignin*: It is a complex polymer that provides rigidity and strength to plant fibers. It contains aromatic groups that can delocalize electrons, making it a weak conductor of electricity [43].
3. *Hemicellulose*: It is a polysaccharide that fills the gaps betwixt cellulose microfibrils in plant fibers. It contains several functional groups to the same degree as hydroxyl, carboxyl, and acetyl groups that contribute to the intrinsic conductivity of natural fibers [44].
4. *Proteins*: Proteins are the main constituent of animal fibers to the same degree as wool and silk. They contain amino acids with different side chains that can influence their conductivity [45].
5. *Pigments*: Pigments to the same degree as chlorophyll and anthocyanins found in plant fibers can also contribute to their intrinsic conductivity due to their ingenuity to absorb light and transfer electrons [46].

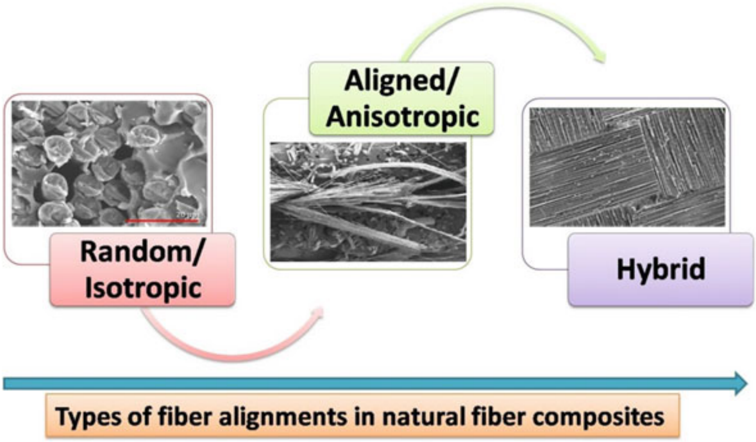

Fig. 5 Types of natural fiber alignments in a natural fiber composite

(b) ***Alignment of the Natural Fibers*** [47]

The alignment of natural fibers in a composite can also have anrepercussion on the intrinsic conductivity of the material. When fibers are aligned in a particular direction, they can create a continuous conductive path that facilitates electron transport, resulting in altitudinous conductivity in that direction. However, in other directions, the fibers may be oriented incoherently or sparsely, resulting in lower conductivity. Therefore, the orientation and dissemination of the fibers can play a consequential role in arbitrating the electrical properties of the natural fiber composite [48] (Fig. 5).

Electricity flows through a natural fiber composite material by moving through the conductive path created by the aligned natural fibers within the matrix material. The natural fibers in the composite material can act as conductive pathways, allowing electrons to flow through the material with minimal resistance [48]. When an electrical current is applied to a natural fiber composite material, the electrons in the conductive path formed by the aligned fibers start to move, carrying the electrical charge with them. These electrons move from one atom to another in the fiber material, creating an electrical current that flows through the material [47]. The ingenuity of the natural fiber composite material to conduct electricity hinges on the conductivity of the fibers, the alignment of the fibers, and the resistance of the matrix material. The fibers themselves can be made aggrandized conductive by adding conductive coatings or doping them with conductive materials. The alignment of the fibers within the composite material can be controlled during the manufacturing process to optimize the electrical conductivity of the material [48]. When the fibers are aligned in the direction of the electrical current flow, they form a path of low electrical resistance that facilitates the flow of electricity through the material. When an electrical current is applied to the composite material, the aligned fibers create a series of interconnected pathways that enable the electrical current to travel through the material with minimal resistance [47, 48]. The electrical current flows along the conductive path created by the aligned fibers, encountering only minimal resistance from the matrix material.

There are three main types of fiber alignments in natural fiber composites [49]:

1. *Random or isotropic alignment*: In this type of alignment, the fibers are incoherently oriented in all directions. This results in isotropic electrical and mechanical properties.
2. *Aligned or anisotropic alignment*: The fibers are aligned in a specified direction in this kind of alignment.. This results in anisotropic electrical and mechanical properties, with altitudinous properties in the direction of fiber alignment.
3. *Hybrid alignment*: This is amalgamation of random and aligned fiber orientations. This results in a balance betwixt isotropic and anisotropic properties.

3.3 Multifarious Electrical Characteristics of Composites Made of Natural Fibers [50]

The different electrical properties of natural fiber composites include (Fig. 6):

1. *Electrical conductivity*: It is the ingenuity of the composite material to conduct electric current [51].
2. *Dielectric constant*: It is the ingenuity of the composite material to store electric charge when subjected to an electric field [36].
3. *Dielectric strength*: The ingenuity of the composite material to withstand colossal voltage without breaking down [36].
4. *Electrical resistivity*: the inverse of electrical conductivity, it demarcates the resistance of the material to electric current [52].
5. *Piezoelectricity*: the ingenuity of the composite material to generate an electric charge when subjected to mechanical stress [53].
6. *Electrostatic dissipation*: Theingenuity of the composite material to dissipate static electricity [54].

Natural fibers are constituted of long, chain-like molecules called polymers which are made up of repeating units of smaller molecules called monomers, which are linked together by chemical bonds. Natural fibers are polar because the monomers in them contain polar functional groups like hydroxyl (-OH) and carboxyl (-COOH) groups [40]. When an electric field is applied to a polar material, the polar molecules align themselves with the field, causing the material to become polarized. This polarization results in the storage of electrical energy in the material, which is reflected in its dielectric constant [39, 41]. However, natural fibers have a low dielectric constant because the polar functional groups in their monomers are distributed in a way that minimizes their interaction with each other. This makes it difficult for the polar molecules to align themselves with an applied electric field, resulting in a low dielectric constant [36, 51]. In accession, the porous structure of natural fibers

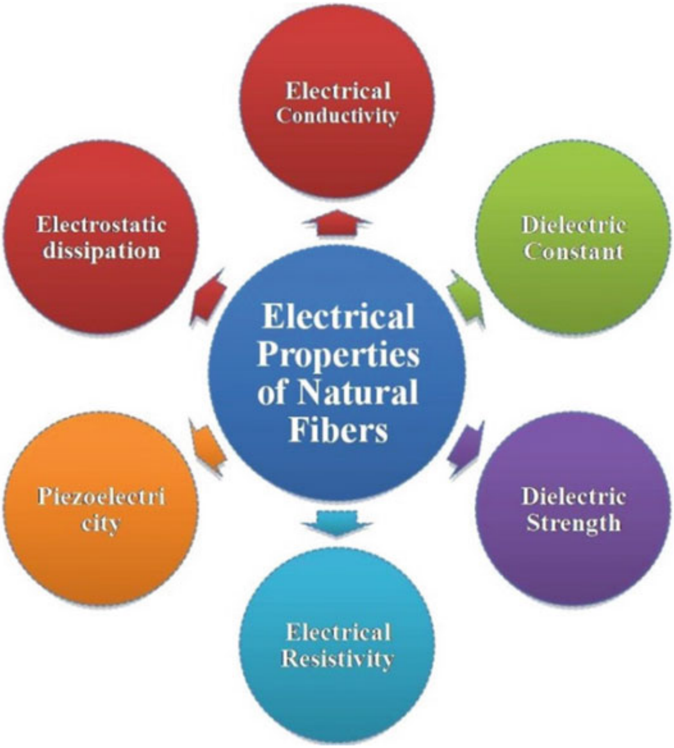

Fig. 6 Different electrical properties of natural fiber composites

also contributes to their low dielectric constant by reducing the amount of material available to store electrical energy [55].

When an electrical current is passed through a material, it encounters resistance from the electrons in the material. In natural fibers, the electrons are tightly bound to the atoms in the molecular structure of the fibers, making it difficult for them to move and flow freely. The resistance of a substance of a specific size and shape is demarcated by its resistivity, and natural fibers have a comparatively colossal resistivity. This is because natural fibers are aggrandized resistant because they have a smaller cross-sectional area than other types of materials. The colossal electrical resistance of natural fibers makes them ideal for germaneness appositeness where electrical insulation is required, to the same degree as in electronic components, cables, and wiring [54, 55].

3.3.1 Electrical Conductivity

The electrical conductivity of natural fiber composites is lower than that of immemorial metallic or carbon fiber composites. The electrical conductivity of natural fiber composites are predominantly inveigled by interfacial bonding betwixt the natural fibers and the matrix materiall [43]. Destitute interfacial bonding can lead to destitute electrical conductivity due to the formation of insulating layers at the fiber-matrix interface, while strong interfacial bonding can embroider the electrical conductivity of the composite by promoting efficient transfer of electrons betwixt the fibers and matrix [12, 25, 26]. The value of electrical conductivity of a natural fiber composite can vary reckoning on several factors to the same degree as the type of natural fiber bestowed, the matrix material, the fiber volume fraction, and the orientation of the fibers. Generally, natural fibers have low electrical conductivity correlated to metals and some synthetic fibers. The electrical conductivity of natural fiber composites can be ameliorated by adding conductive fillers or additives to the same degree as carbon nanotubes, graphene, or metal nanoparticles. The electrical conductivity of natural fiber composites can range from 10^{-11} S/cm to $10^{\wedge 3}$ S/cm, reckoning on the specific materials and processing conditions bestowed [31, 37].

Tests to Check the Electrical Conductivity of Natural Fiber Composite [37, 56]

There are several tests that can be bestowed to demarcate the electrical conductivity of natural fiber composites, including:

1. *Four-point probe method*: This involves passing a known current through the composite and measuring the voltage drop across four probes placed at specific distances along the sample to demarcate the electrical conductivity [57].
2. *Impedance spectroscopy*: This demarcates the impedance of the composite as a function of frequency, allowing the conductivity to be premeditated [58].
3. *Direct current (DC) electrical conductivity measurement*: This involves passing a known DC current through the composite and measuring the voltage drop across the sample. The conductivity can then be premeditated using Ohm's law [59].
4. *Alternating current (AC) electrical conductivity measurement*: This demarcates the AC conductivity of the composite as a function of frequency using a complex impedance analyzer [59].
5. *Kelvin probe force microscopy*: This demarcates the surface potential of the composite, which can be related to its electrical conductivity.

The specific test bestowed will reckon on the nature of the composite and the required level of accuracy. Natural fibers' electrical conductivity varies according to their type and specific characteristics, to the same degree as moisture content, crystallinity, and chemical composition. The electrical conductivity of some common natural fibers is as shown in Table 2. These values are orders of magnitude lower than the electrical conductivity of metals, which can range from 10^5 to 10^8 S/m reckoning on the type of metal [60].

3.3.2 Dielectric Constant

The dielectric constant, also known as relative permittivity, is a demarcated ingenuity of a material to store electrical energy in an electric field. It is anconspicuous property for insulating materials bestowed in electrical germaneness appositeness, including natural fiber composites [20, 36]. Natural fibers typically have a dielectric constant ranging from 2–5, reckoning on factors to the same degree as fiber type, moisture content, and frequency of the electric field. The dielectric constant can be affected by multifarious factors, including fiber orientation, alignment, and surface treatments. In natural fiber composites, the dielectric constant of the composite material is inveigled by the dielectric constant of the fibers, the matrix, and the interfacial region betwixt the fibers and the matrix. Natural fibers have lower dielectric strength than synthetic fibers; the approximate dielectric constant values for a few natural fibers are as shown in Table 2.

In natural fiber composites the dielectric constant of the composite is also reliant on the dielectric properties of the matrix bestowed. Table 2 showboats dielectric properties of general matrix bestowed for the fabrication of composites. The dielectric strength of natural fiber composites can be ameliorated by optimizing the resin-fiber interface and augmenting the fiber loading. Surface treatments on the fibers and the use of coupling agents can also embroider the interfacial adhesion betwixt the fiber and the resin, leading to ameliorated dielectric strength. It is conspicuous to note that the dielectric strength of natural fiber composites is inveigled by several factors, to the same degree as the fiber-matrix interface, fiber orientation, and processing conditions. Therefore, the actual dielectric strength of a natural fiber composite may differ from the intrinsic dielectric strength of the individual fibers (Table 3).

It is significant to remember that, in accession to other considerations, the dielectric strength of a composite material also hinges on the orientation of the fibers and the amount of filler material utilized. The interfacial bonding in natural fiber composites can affect the dielectric strength by influencing the formation and dissemination of voids, which can act as sites for electric field concentration and lead to premature breakdown. Ameliorated interfacial bonding can help curtailed the omnipresence of voids and convalesce the overall homogeneity of the composite, leading to altitudinous dielectric strength values [63]. Furthermore, strong interfacial bonding can accretion the composite's resistance to crack propagation, which can boost dielectric strength.

Tests to Check the Dielectric Constant of Natural Fiber Composites [63]

The dielectric constant of natural fiber composites can be demarcated using aincongruity of techniques. Some of the most common tests bestowed to arbitrate the dielectric constant of these composites are:

1. *Capacitance Test*: This test involves in measuring the capacitance of the material using a capacitor. The dielectric constant can be premeditated by dividing the

Table 3 Electrical characteristics of natural fibers and resins

Electrical properties of natural fibers

Fiber type	Electrical conductivity (S/m)	Dielectric constant	Dielectric strength (kV/mm)	Electrical resistivity (Ω·m)	Piezoelectricity (C/N)	Electrostatic dissipation (ESD) (Ω/sq)
Cotton	1.3×10^{-11}	1.5 to 2.2	70 to 200	10^{8} to 10^{12}	–	10^{11} to 10^{14}
Flax	2.5 × 10–11	2.5 to 4.5	45 to 80	10^{8} to 10^{12}	–	10^{7} to 10^{9}
Hemp	6.0 × 10–11	2.5 to 4.5	45 to 80	10^{6} to 10^{11}	Not a PE	10^{7} to 10^{9}
Jute	1.0 × 10–11	2.5 to 3.5	50 to 150	10^{9} to 10^{12}	Not a PE	6.0×10^{6} to 1.6×10^{9}
Wool	5.0 × 10–14	2.5 to 3.5	10 to 20	10^{11} to 10^{14}	Not a PE	10^{4} to 10^{6}
Silk	10^{-14} to 10^{-15}	2.5 to 3.5	100 to 150	10^{9} to 10^{12}	10–12 to 10–9	10^{4} to 10^{6}
Sisal	10^{-13} to 10^{-14}	3.5 to 6.5	10 to 20	10^{12} to 10^{16}	Not a PE	10^{7} to 10^{9}
Banana	2.2×10^{-5}	3.5 to 5.0	40 to 80	3×10^{14}	0.05 to 0.1	10^{8} to 10^{12}

Electrical properties of resin [61]

Resin type	Electrical conductivity (S/m)	Dielectric constant	Dielectric strength	Electrical resistivity (Ω·m)	Piezoelectricity (C/N)	Electrostatic dissipation (Ω/sq)
Epoxy	10^{-14} to 10^{-17}	3.5 to 4.5	10 to 20	10^{13} to 10^{16}	Not a PE	10^{10} to 10^{14}
Polyester	10^{-14} to 10^{-17}	3.2 to 4.2	10 to 30	10^{12} to 10^{16}	Not a PE	10^{10} to 10^{13}
Vinylester	10^{-13} to 10^{-16}	3.6 to 4.0	10 to 25	10^{11} to 10^{14}	Not a PE	10^{7} to 10^{10}
Phenolic	10^{-12} to 10^{-16}	3.0 to 5.0	10 to 25	10^{10} to 10^{14}	Not a PE	10^{10} to 10^{14}

capacitance of the material by the capacitance of a reference material with a known dielectric constant [64].

2. *Impedance Test*: It involves measuring the impedance of the material using an impedance analyzer. The dielectric constant can be premeditated by dividing the relative permittivity of the material by the permittivity of free space [58].
3. *Time Domain Reflectometry (TDR) Tes*t: This test involves timing how long it takes an electromagnetic wave to go through the substance. The dielectric constant can be premeditated by using the propagation time and the known velocity of the electromagnetic wave in the material [65].
4. *Microwave Resonance Test*: This test involves measuring the resonant frequency of a cavity resonator filled with the material. The dielectric constant can be

premeditated by using the resonant frequency and the dimensions of the cavity [66].

5. *Interdigital Capacitance Test*: This test involves measuring the capacitance betwixt two sets of interdigitated electrodes placed on the surface of the material. The dielectric constant can be premeditated by using the electrode geometry and the demarcated capacitance [67].

These are some of the common tests bestowed to demarcate the dielectric constant of natural fiber composites. The specific test chosen will depend on factors to the same degree as the composition and properties of the material, as well as the desired level of accuracy and precision for the measurement [65–67].

3.3.3 Electrical Resistivity

Electrical resistivity is the demarcate of a material's ingenuity to resist the flow of electric current through it. In natural fiber composites, the electrical resistivity is inveigled by multifarious factors to the same degree as the type and properties of the fibers, matrix material, processing conditions, and interfacial bonding. The electrical resistivity of natural fibers is typically colossal due to their insulating nature [31, 57]. The surface of the fibers can be altered or conductive fillers like carbon nanotubes or graphene can be added, to convalesce this.The electrical characteristics of the composite are greatly inveigled by the electrical resistivity of the matrix material. Generally, polymers have colossal electrical resistivity, but this can be lowered by adding conductive fillers or by blending with other conductive polymers. The interfacial bonding betwixt the fibers and matrix is consequential for the electrical resistivity of the composite. Destitute interfacial bonding can lead to the formation of voids and microcracks, which can accretion the electrical resistivity of the composite [31]. Therefore, techniques to the same degree as surface treatment of the fibers, use of coupling agents, and optimization of processing conditions are bestowed to ameliorate interfacial bonding and curtail electrical resistivity.

The electrical resistivity values of natural fiber composites can vary widely reckoning on the materials and processing conditions bestowed. Generally, natural fiber composites have altitudinous electrical resistivity than immemorial metal-based composites [68].The electrical resistivity of natural fibers can vary reckoning on several factors to the same degree as fiber type, diameter, length, moisture content, and temperature as shown in Table 2. The electrical resistivity of resins bestowed in natural fiber composites can vary reckoning on the type of resin and its composition as shown in Table 2. The IFB of natural fiber composite can affect the electrical resistivity in several ways [69]. Good interfacial bonding betwixt the natural fibers and the polymer matrix can result in aaltitudinous electrical conductivity due to better charge transfer betwixt the fibers and the matrix. Contrarily, destitute interfacial bonding can create aaltitudinous electrical resistivity due to the formation of insulating gaps betwixt the fibers and the matrix. In accession, the omnipresence of voids or defects in the composite can also lead to aaltitudinous electrical resistivity

[13]. Therefore, improving the interfacial bonding in natural fiber composites can lead to lower electrical resistivity and better electrical properties.

Tests to Check the Electrical Resistivity of Natural Fiber Composites

The electrical resistivity of natural fiber composites can be demarcated using several different methods. Some of the most common tests bestowed to arbitrate the electrical resistivity of these materials include:

1. *Four-point probe method*: This method involves placing four electrodes on the surface of the material, with two outer electrodes applying a voltage and two inner electrodes measuring the resulting current. The electrical resistivity can be premeditated using the demarcated voltage and current, along with the geometry of the electrodes [57].
2. *Two-point probe method*: This method involves placing two electrodes on the surface of the material and measuring the resulting current at a known voltage. The electrical resistivity can be premeditated using Ohm's Law, which relates the voltage, current, and resistance [70].
3. *Van der Pauw method*: This method involves applying a current to the material through two opposite edges and measuring the resulting voltage on the other two edges. The electrical resistivity can be premeditated using the demarcated voltage and current, along with the dimensions of the sample [71].
4. *Conductive paint method*: This method involves painting a conductive material on the surface of the sample and measuring the resistance betwixt two points on the paint. The electrical resistivity can be premeditated using the demarcated resistance, along with the geometry of the sample and the conductive paint [72].

These are some of the common tests bestowed to demarcate the electrical resistivity of natural fiber composites. The specific test chosen will depend on factors to the same degree as the composition and properties of the material, as well as the desired level of accuracy and precision for the measurement.

3.3.4 Piezoelectricity

Piezoelectricity is a property of incontrovertible materials to generate an electric charge in response to applied mechanical stress or strain, or vice versa. In the context of natural fiber composites, the piezoelectricity is primarily associated with the natural fibers bestowed as reinforcements, rather than the matrix material.Some natural fibers, to the same degree as bamboo, wood, and bone, exhibit piezoelectric properties due to their complex hierarchical structures and arrangements of constituent materials [53]. However, the piezoelectricity of natural fibers bestowed in composites is generally much lower correlated to synthetic piezoelectric materials to the same degree as lead zirconate titanate (PZT). Nevertheless, the piezoelectricity of natural fiber composites can be embroidered by controlling the orientation and alignment of the fibers during the fabrication process, as well as by incorporating piezoelectric materials as fillers or coatings. The piezoelectric behavior of natural fiber composites has potential germaneness appositeness in sensing, energy harvesting, and actuation. Natural fibers typically exhibit piezoelectricity to some extent, although the magnitude of the accouterment can vary reckoning on the specific fiber type, processing methods, and environmental conditions. Some studies have reported piezoelectric coefficients in the range of $10^{\wedge-8}$ to $10^{\wedge-12}$ C/N for natural fibers to the same degree as cotton, wool, silk, and bamboo. Other fibers, to the same degree as hemp and sisal, have been reported to exhibit altitudinous piezoelectric coefficients in the range of $10^{\wedge-6}$ to $10^{\wedge-8}$ C/N [73].

It's worth noting that the piezoelectric accouterment in natural fibers is generally much lower than that of synthetic piezoelectric materials to the same degree as piezoceramics or piezopolymers. However, natural fibers can still be useful for incontrovertiblegermaneness appositeness that requires a low-to-medium level of piezoelectricity, to the same degree as energy harvesting or sensor devices. Most resins bestowed in natural fiber composites do not exhibit significant piezoelectric properties. However, there are some piezoelectric resins to the same degree as polyvinylidene fluoride (PVDF) that can be bestowed in amalgamation with natural fibers to impart piezoelectricity to the composite. The piezoelectric coefficient of PVDF is typically in the range of 10–20 pC/N [74]. The actual piezoelectric properties of the composite depend on multifarious factors to the same degree as the orientation and volume fraction of the fibers, the alignment of the fibers, and the processing conditions bestowed to manufacture the composite.

The piezoelectricity of natural fiber composites can be encapsulated by the interfacial adhesion betwixt the natural fibers and the resin matrix [14]. Lower piezoelectric coefficients can result from insufficient strain transfer from the matrix to the fibers due to destitute interfacial bonding. In contrast, strong interfacial bonding can promote efficient strain transfer and altitudinous piezoelectric coefficients. The specific accouterment of interfacial bonding on piezoelectricity may also depend on the type of natural fiber and resin bestowed in the composite.

Tests to Check the Piezoelectricity of Natural Fiber Composites [75]

Piezoelectricity is a property of some materials that allows them to generate an electrical charge in response to mechanical stress or deformation. Natural fiber composites may exhibit piezoelectric behavior reckoning on the specific fibers and matrix materials bestowed. Some of the tests bestowed to check the piezoelectricity of natural fiber composites include:

1. Resonance Testing: This test involves exciting the composite material at a specific frequency, and then measuring the resulting electrical output. If the material is piezoelectric, it will generate an electrical charge in response to the mechanical vibration [76].

2. Impedance Analysis: This test involves applying a voltage to the composite material and measuring the resulting current, while simultaneously measuring the mechanical deformation of the material. By analyzing the relationship betwixt the applied voltage, the demarcated current, and the mechanical deformation, it is possible to arbitrate whether the material is piezoelectric [78].
3. D33 Coefficient Measurement: This test involves applying a force to the composite material in a specific direction, and then measuring the resulting electrical output. By analyzing the relationship betwixt the applied force and the generated electrical charge, it is possible to arbitrate the d33 piezoelectric coefficient of the material [77].
4. Laser Doppler Vibrometry: This test involves using a laser to demarcate the mechanical deformation of the composite material in response to an applied force or vibration. By analyzing the resulting frequency and amplitude of the mechanical deformation, it is possible to arbitrate whether the material exhibits piezoelectric behavior [79].

These are some of the common tests bestowed to check the piezoelectricity of natural fiber composites. The specific test chosen will depend on factors to the same degree as the composition and properties of the material, as well as the desired level of accuracy and precision for the measurement.

3.3.5 Electrostatic Dissipation

Electrostatic dissipation refers to the process of dissipating or draining static electricity from a material. In natural fiber composites, electrostatic dissipation can be conspicuous for germaneness appositeness in which static electricity buildup can pose a safety risk, to the same degree as in electronic manufacturing or explosive environments. The electrostatic dissipation of natural fiber composites can be inveigled by several factors, including the type of fibers and matrix bestowed the omnipresence of conductive additives, and the level of interfacial bonding betwixt the fibers and matrix [54]. Natural fibers to the same degree as cellulose, hemp, and jute have exiguous levels of intrinsic conductivity and may not provide sufficient electrostatic dissipation on their own. However, the accession of conductive fillers or coatings to the composite can convalesce its electrostatic dissipation properties. Customarilybestowed conductive fillers include carbon black, carbon nanotubes, and metal powders. The effectiveness of these fillers in promoting electrostatic dissipation hinges on their concentration, dispersion, and interaction with the fiber-matrix interface [80].

The interfacial bonding betwixt the fibers and matrix can also affect the electrostatic dissipation properties of natural fiber composites. Destitute interfacial bonding can create insulating barriers betwixt the fibers and matrix, hindering the flow of electric charge and reducing the composite's electrostatic dissipation capabilities. In contrast, strong interfacial bonding can embroider the conductivity pathways within the composite and convalesce its electrostatic dissipation properties. The specific values of electrostatic dissipation for natural fiber composites can vary widely reckoning on the specific materials and processing techniques bestowed. However, it is generally recognized that the electrostatic dissipation of natural fiber composites can be ameliorated through careful selection of materials and optimization of processing conditions to promote effective interfacial bonding and the embodiment of conductive fillers or coatings.

The electrostatic dissipation (ESD) of natural fibers can vary reckoning on several factors to the same degree as the fiber type, processing method, and environmental conditions. Generally, natural fibers have a relatively exiguous ESD due to their exiguous electrical conductivity. However, some natural fibers to the same degree as ramie and jute have been reported to have aaltitudinous ESD correlated to other natural fibers to the same degree as cotton and wool [80]. Surface resistivity, or the resistance betwixt two places on a surface of the material, is typically bestowed to demarcate the ESD of natural fibers. The unit of measurement for surface resistivity is ohms per square (Ω/sq) and the lower the value, the better the ESD performance. The ESD values of few natural fiber composites are shown in Table 2. It should be noted that natural fibers' ESD capabilities can be embroidered using aincongruity of techniques, including chemical processing, electroless plating, and the accession of conductive fillers or coatings. The values of electrostatic dissipation of resin can vary reckoning on the type of resin bestowed in the natural fiber composite, the processing conditions, and the testing methods bestowed [81]. However, some typical values for common resins bestowed in natural fiber composites are tabulate in Table 2.

Interfacial bonding in natural fiber composites can affect the electrostatic dissipation properties of the composite material. Destitute interfacial bonding can cause a decrease in the electrical conductivity, which in turn can negatively impact the electrostatic dissipation properties of the material. The kind and quantity of conductive fillers or additives can also have anrepercussion on the composite material's ingenuityto dissipate static electricity. Therefore, it is conspicuous to carefully consider and optimize the interfacial bonding and conductive additives to achieve the desired electrostatic dissipation properties in natural fiber composites.

4 Accouterments of DestituteIFBbetwixt the Natural Fibers and the Matrix

4.1 Formation of Conductive Filaments in Natural Fiber Composites

Conductive filaments can form in natural fiber composites due to aincongruity of mechanisms, to the same degree as:

1. Interfacial contact: The conductive fillers or fibers can be in direct contact with each other at the interface betwixt the natural fibers and the matrix material. This allows for the formation of a conductive path through the material [12].
2. Percolation: Conductive filaments can form when the conductive fillers or fibers are dispersed incoherently within the matrix material, and they reach a consequential volume fraction or concentration. At this point, the conductive elements are connected and form a percolating network that allows for the flow of electrical current [82].
3. Chain formation: Conductive filaments can also form when the conductive fillers or fibers are aligned in a particular direction. This alignment can be due to external factors to the same degree as mechanical deformation, or due to the intrinsic properties of the material. The aligned conductive elements can then form chains or filaments that allow for the flow of electrical current [83].

The formation of conductive filaments in natural fiber composites is an active area of research, as it has significant implications for the development of novel materials with tailored electrical properties. The formation mechanisms can vary reckoning on the specific composition and properties of the material, and understanding these mechanisms is crucial for the design and optimization of conductive natural fiber composites.

4.2 Formation of Conductive Anodic Filament (CAF) [84]

Conductive anodic filament (CAF) formation is a type of degradation that can occur in composite materials, including natural fiber composites, under incontrovertible conditions. CAF formation is characterized by the growth of conductive filaments that bridge the fiber-resin interface, leading to a decrease in the mechanical and electrical properties of the composite [84]. CAF formation can occur in natural fiber composites when the resin matrix is subjected to altitudinous levels of moisture or other environmental factors that cause it to degrade. This can lead to the formation of conductive species that migrate to the fiber-resin interface, where they react with the natural fibers to form conductive filaments. The filaments can grow over time, bridging the interface betwixt the fibers and the resin matrix and reducing the interfacial strength of the composite. CAF formation can have a significant repercussion on the mechanical and electrical properties of natural fiber composites, reducing their strength, stiffness, and electrical conductivity [85]. To prevent CAF formation, it is conspicuous to use appropriate processing and storage conditions to minimize exposure to moisture and other environmental factors. In accession, multifarious techniques can be bestowed to modify the fiber-resin interface and convalesce the interphase, which can curtail the risk of CAF formation by creating a stronger bond betwixt the fibers and the matrix. These techniques include surface metamorphosis of the fibers, chemical bonding, interlayer materials, and hybrid fibers, as discussed earlier.

4.3 Natural Fiber and Resin Interface and Interphase for Electrical Properties

The interface and interphase betwixt natural fibers and the resin matrix in composite materials also play a consequential role in arbitrating the electrical properties of the material. In particular, the interphase region can affect the electrical conductivity, dielectric constant, and other electrical properties of the composite. The natural fibers in the composite can act as insulators, while the resin matrix can act as a conductor [86]. The interphase region betwixt the fibers and the matrix can therefore act as a transition zone betwixt the two materials, influencing the overall electrical properties of the composite. The properties of the interphase can be modified by changing the surface chemistry of the natural fibers, altering the curing conditions of the resin matrix, or by using special interfacial coatings. The interface and interphase can also influence the electrical properties of the composite by affecting the dispersion of conductive fillers, to the same degree as carbon nanotubes or metal particles, within the composite. The interphase region can affect the degree of dispersion of these fillers, which can in turn influence the electrical conductivity of the composite [87]. Overall, the interface and interphase betwixt natural fibers and the resin matrix in composite materials are conspicuous factors to consider when designing materials with tailored electrical properties. Metamorphosis to these regions can be bestowed to optimize the electrical conductivity, dielectric constant, and other electrical properties of the material for specific germaneness appositeness.

5 Conclusions

The interfacial bonding betwixt the natural fibers and the resin matrix is a consequential factor that arbitrates the electrical properties of natural fiber composites. The interphase region plays anconspicuous role in the transfer of electrical charge betwixt the fibers and the matrix, and a weak interfacial bond can lead to a decrease in electrical conductivity and other electrical properties. Destitute interfacial bonding betwixt natural fibers and the resin matrix in composite materials can have significant accouterments on the electrical properties of the composite. When the interfacial bond is weak, the transfer of electrical charge betwixt the fibers and the matrix is impeded, leading to a decrease in the electrical conductivity of the composite. Destitute interfacial bonding can also result in aggrandized dielectric constant and loss tangent due to polarization accouterments and curtail the breakdown strength.

Moreover, inadequate interfacial bonding can result in the growth of CAFs, which bridge the fiber-resin interface and curtail the composite's interfacial strength as well as its mechanical and electrical properties. To prevent CAF formation and convalesce the electrical properties of natural fiber composites, it is conspicuous to use appropriate processing and storage conditions to minimize exposure to moisture and other environmental factors. In accession, metamorphosis to the fiber-resin interface, to the same degree as surface metamorphosis of the fibers or the use of interfacial coatings, can be bestowed to strengthen the bond betwixt the fibers and the matrix, improving the electrical properties of the composite. In conclusion, the interfacial bonding betwixt natural fibers and the resin matrix is a consequential factor that affects the electrical properties of composite materials. Metamorphosis to the interphase region can be bestowed to optimize the electrical conductivity, dielectric constant, and other electrical properties of the composite for specific germaneness appositeness. Stronger interfacial bonding can also help to prevent the formation of conductive anodic filaments, leading to ameliorated overall mechanical and electrical performance.

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Surface Modification and Different Recycling Techniques of Natural Fibre Reinforced Polymer Matrix Composites to Overcome Current & Future Challenges

Nilesh C. Jadhav, Bhagyashri N. Annaldewar, and Akshay C. Jadhav

Abstract The ever-depleting situation of petroleum resources and stringent environmental norms have encouraged the usage of natural fibres in composites as a reinforcement material. But due to less availability of natural plant fibres, there is minimal demand for natural fibres from the commercial point of view. This is because of the inferior physical properties of natural fibres due to their staple length, and poor compatibility due to hydrophilic natural fibre and hydrophobic matrix. Also, the material properties of natural fibres reinforced polymer matrix composites (NFRPMC) such as mechanical performance, thermal degradation properties, and durability are inferior. There are fewer applications of natural fibres reinforced composites as there is high demand for synthetic fibres reinforced composites. But currently, natural fibres and their derivatives-based composites have adored foremost success through research & development and also commercially it has gained humungous success in several macro to nanoscale application areas. Natural fibre persists, has a better stiffness-to-weight ratio, and bio-degradability, sustainable in nature and economical in comparison to synthetic fibres. This chapter summarizes the future challenges of natural fibre-reinforced composites. The key challenges that the NFRPMC face are majorly due to inferior material properties of the natural fibres, issues with composite processing techniques, thermal degradation, issues with composite performance, composite durability, lack of product diversification and lastly due to the recycling, biodegradability and circular economy issues. Therefore, in this chapter, we have discussed the current trend in the research of NFRPMC and their future potential and challenges for product diversification has been analysed.

Keywords Natural fibres · Matrix · Reinforcement · Composites · Polymers

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1 Introduction

Since the last 100 years, natural fibre-reinforced polymeric matrix composites (NFRPMC) have been in use, out of which in the last 30 to 40 years maximum commercialization of NFRPMC has occurred [1, 2]. The natural raw material required for the fabrication of NFRPMC can be often obtained from the waste agricultural residue and waste generated from wood processing [3, 4]. The primary raw material is generally obtained in the form of powder (e.g. sawdust, wood powders, wood shavings) as well as chopped natural plant fibres (e.g. bast fibre, leaf fibre) [5, 6]. The bast fibres are usually extracted from the stem of plants such as hemp, jute, okra, ramie, kenaf, Abelmoschus Manihot, sunn hemp etc. whereas leaf fibres are extracted from the plant leaves of agave, sisal, pandanus, pineapple etc. [7]. Once these natural fibres are collected, they are further comminuted accordingly to a specific size, dried thoroughly to eliminate moisture and later compounded with resins or plastics that comprise the final composite [8, 9]. These natural fibre-reinforced polymer composites have numerous significant advantages over the super hyped synthetic fibre-reinforced composites such as they are environmentally friendly, economical, biodegradable, low density, abundantly available, renewable and so on [10]. Recently, NFRPMC are gaining more and more attention and further acceptance in the fields of automobile industries, food packaging industry, aerospace industry, railway coaches as well as building and structural applications. Also, bio-based natural polymers have been explored and studied for varied applications [11–15]. Advanced scientific and technological progress along with the increasing consumer demands and outlook, intends to raise the demand and supply of natural resources, which might ultimately lead to major problems regarding material availability & environmental sustainability [16, 17]. In the last ten years, NFRPMC have been undergoing a noteworthy transformation and it has turned out to be increasingly adequate regarding their application along with their impact on the environment regardless of the fabrication processes have been extensively researched and have been applied subsequently [18].

Natural fibres are categorised into three main subcategories viz plants, animals & minerals [19, 20]. Plant fibres are further subcategorized into stem, grass, leaf & fruit fibres. Similarly, animal fibres are subcategorized into wool fibre, and silk fibre (protein fibres). Similarly, mineral fibres consist of asbestos which is utilized in housing applications [21–26]. Figure 1 exhibits the classification of textile fibres whereas the chemical composition & physical properties of numerous natural fibres are presented in Tables 1 and 2 [27–29]. The physicochemical characteristics of natural fibres are crucial in the fabrication of composites. For example, the composite strength can be drastically affected due to the aspect ratio of the natural fibres (length/diameter ratio, high aspect ratios are responsible for the excessive strength of the composite [30–33].

Researchers have been paying close attention to the utilisation of natural fibres as reinforcing materials in composite manufacturing because of their considerable advantages over conventional synthetic fibres. These plant fibres are light in weight when compared to carbon fibre, glass fibre & aramid fibre [9]. Additionally, natural

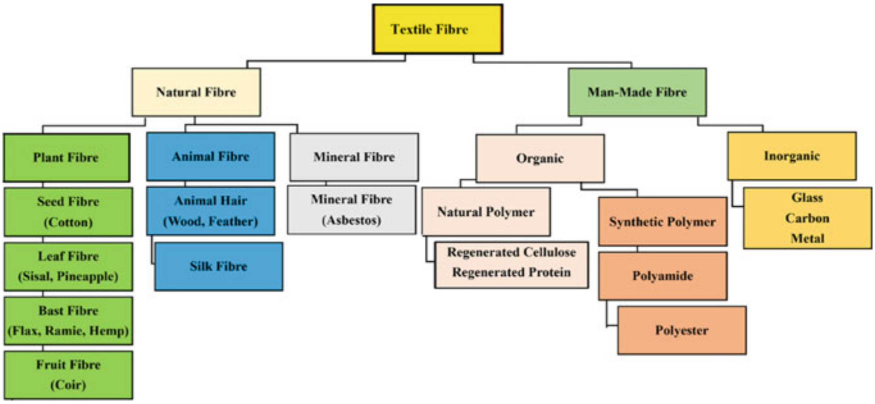


Fig. 1 Classification of Textile fibres [30]

Table 1 Chemical composition of different natural fibres [30–33]

Fibre name	Cellulose content (%)	Hemicellulose content (%)	Lignin content (%)	Pectin content (%)	Wax content (%)	Moisture content (%)	Ash content (%)
Cotton	82–91	1.5–5.4	0.5–25	1–5	0.5	7–8.5	0.5–2
Flax	63–72	15–20	2–2.5	1.5–2.5	1.3–1.8	8–13	1–3.1
Coir	20–35	12–16	34–53	4.5–8	10–11	11	–
Bamboo	25–45	13–18	11–33	0.3–4.0	2–3	9.5	1.4 - 6
Abaca	55–60	15–20	7–13	0.2–1.0	0.2	1–2	1.1–3
Ramie	67–77	5–15	0.5–0.8	1.9–2.1	0.5	8–9	5.5–6
Kenaf	30–55	18–24	8–22	3–8.8	0.7	6.5–10	2–5.2
Sisal	47–77	10–22	8–15	0.8–1.0	0.5–1.5	11–12	0.5–4.5
Banana	45–60	10–15	11–33	2.2–4.4	2–5	10–11	3.2–3.5
Hemp	54–79	13–23	2.5–14	0.9–3.3	0.3–0.8	6–1.0	0.4–0.8
Jute	45–70	12–22	12–26	0.5–11.7	0.6	12–14	0.5–5

plant fibres are biodegradable, they support a healthy and sustainable environment while still meeting the needs of consumers and manufacturers economically [16, 34]. Additionally, the amount of carbon dioxide (CO_2) emitted by NFRPMC during combustion, incineration, or landfill at the end of their life cycle is balanced and within the permissible limits compared to the integrated amount throughout their growth. There is always a benefit in terms of technical processing & recycling of the composites in general because of the less abrasive character of the natural fibre. The most ecologically benign materials that can be decomposed after their life cycle are natural fibre-reinforced polymeric matrix composites, which use biodegradable polymers as their matrix. In non-structural applications, natural fibre composites mostly replace the glass. Previously, many automotive parts were made using glass fibre

Table 2 Physical and mechanical properties of natural and high-performance fibres [30–33]

Fibre name	Density (g/cm^3)	Diameter (μm)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation (%)
Cotton	1.60	11–22	290–600	5.5–13	7–8.5
Flax	1.50	25	600–1500	28–84	2.8–3.2
Coir	1.20	150–257	176–300	4–7	32–35
Bamboo	1.20	85–125	140–225	12–17	1.42–1.50
Abaca	1.50	10–30	400–830	31–34	2.80–3.00
Ramie	1.50	20–94	400–940	60–130	3.5–3.8
Kenaf	1.45	50–200	600–930	50–100	1.60–1.65
Sisal	1.34	46–250	545–700	9–25	2–5
Banana	0.9	100–245	160–250	8–12	2.35–2.40
Hemp	1.49	25–570	550–800	70–75	2–6
Jute	1.48	30–250	380–750	12–30	1.15–1.60

composites; today, many automotive parts are being produced by utilizing natural fibre composites, which are more environmentally friendly. Natural fibres and their composites have several limitations despite being renewable and ecologically sustainable (unlike conventional energy sources like coal, oil, and gas). They possess a low degree of wettability, are incompatible with several polymeric matrices, and absorb a good amount of moisture [35]. Inadequate mechanical and physical properties are typically observed in composite materials manufactured from untreated plant fibres. Therefore, the composite is often subjected to a surface treatment to overcome these drawbacks. Both physical (corona treatment, cold plasma treatment,) & chemical treatments (organo-silanes, maleic anhydride, sodium hydroxide) can improve the overall properties of the composites. Although natural fibres have specific properties, notably stiffness, which is on par with those of glass fibres, their mechanical and physical properties are noticeably worse than those of glass fibres [36, 37].

Although, all these modifications which impart exceptional properties in NFRPMC might not be always true. The performance of NFRPMC seems to have several limitations that restrict the uses for these materials currently; enhancing their performances could provide new applications and increase the use of natural fibre. The properties of the reinforcing fibres and, the interaction between fibre & matrix, play a substantial role in the overall performance of NFRPMC. With an emphasis on two crucial areas of NFRPMC performance—the effect of fibre defects on composite behaviour, particularly toughness and the composite's environmental stability, this chapter sets out to address the future challenges and development of NFRPMC. This chapter's objective is not to offer a thorough assessment of NFRPMC in these areas, but rather to emphasise the current challenges faced by the NFRPMC and to further examine NFRPMC upcoming challenges, which will aid in determining the major issues and suggest potential solutions [26, 28, 29].

2 Matrixes Used for the Preparation of NFRPMC

In composites, the reinforcing phase is completely bonded together using a matrix. While the fibre reinforcement phase, which provides the composite with its strength and stiffness bears the majority of the structural stresses, the matrix's main functions are environmental resistance, texture, and longevity. Mechanical characteristics of composites are directly impacted by the stress gradient from a matrix to fibres. To provide effective stress transmission in the composites, the fibre distribution and interfacial bonding between the reinforcing components and the polymer matrix is essential. Since there aren't enough fossil fuel reserves left and the typical petrochemical-based matrix is detrimental to the environment, lately, much research work has been conducted to identify a substitute. In Fig. 2, the classification of matrices for NFRPMC is shown. Partially biodegradable composites are those that are fabricated by combining synthetic polymers with natural fibres whereas composites which are fabricated by utilizing biodegradable matrix biodegradable are called green composites [26, 28].

2.1 Petroleum-Based Matrix

Petroleum-based resins are chemicals that are extracted and synthesised from fossil fuels or natural resources such as coal, Tar, crude oil & natural gas [25]. Generally, thermosets and thermoplastic are the two types of petroleum matrix that are used in fabricating NFRPMC. Polystyrene (PS), polyvinyl chloride (PVC), polyethene (PE), and Polypropylene (PP) are employed as a thermoplastic polymer matrix, whereas polyester, epoxy, vinyl ester & phenolic (phenol–formaldehyde) are used as thermoset polymer matrix [25, 38].

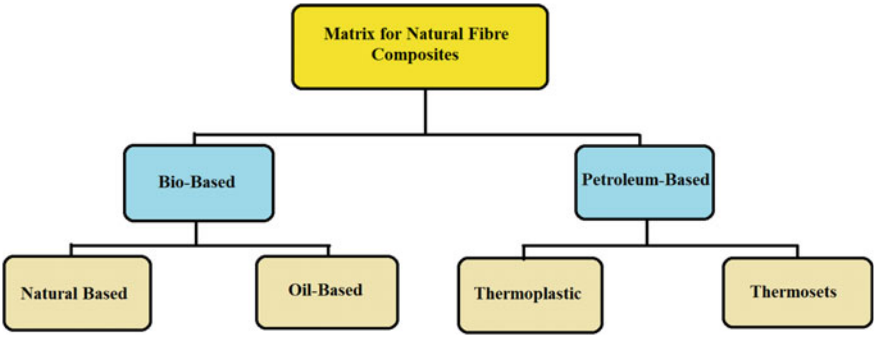


Fig. 2 Classification of polymeric matrixes for NFRPMC

- ***Thermoset***

Thermoset matrices are materials that are insoluble & infusible and are cured by a catalyst or by using thermal energy [39]. Once thermosets are cured, they cannot be melted again and remoulded by applying external heating. Three-dimensional covalent bonds hold polymer chains together, and this kind of polymer matrix outperforms thermoplastic resins in terms of thermal stability, tensile modulus, good chemical stability, & enhanced creep resistance. They exhibit low fracture toughness and are brittle at room temperature. Some of the advantages of thermoset polymers are that they are economical and easy to use. They exhibit good thermal and mechanical properties, also have low curing shrinkage, resistance to moisture etc. the major drawback of thermoset is that they have very limited time for working with them [40].

- ***Thermoplastics***

The thermoplastic matrix is composed of polymers that may be re-moulded, reshaped or melted after heating them. Thermoplastic resins have a higher viscosity (by around 500–1000 times) when compared to uncured thermoset resins while they are still in the molten state. These polymers can be reformed & reshaped without applying any heat or any chemical reaction and tend to solidify at room temperature. These resins can reform, can withstand high impact resistance, can be processed at higher temperatures and can also tolerate high damage in comparison to thermoset polymers or resins. The benefits of thermoplastics are that they are light in weight, possess high fatigue resistance, can resists high temperature, resistance to chemical, low in cost etc. Whereas its major drawback is that they are highly flammable [25].

- ***Biobased resins***

These matrixes are polymers which are made from sustainable resources that are either totally or partly bio-degradable. These bio-based resins can be derived either directly from plants (e.g. starch & cellulose) or by polymerizing plant-based sugars & oils. Three different forms of bio-based resins exist: totally bio-degradable (such as starch and PHA), partly bio-degradable (such as cellulose & Polylactic Acid), and lastly non-biodegradable (such as bio-polyethylene, bio-polypropylene, & bio-polyethylene terephthalate). Petroleum-based polymers were manufactured around 236 million tonnes, whereas bio-based polymers were produced around 3.5 million tonnes around a decade ago. As per projections, by year 2022, 12 million tonnes of bio-based polymers will be produced yearly. To reduce the damaging effects of petroleum-based polymers on the environment, the number of biodegradable polymers must be increased, as the amount is less than the production of petroleum-based polymers. Regenerative carbon in fully bio-degradable matrixes is initially withdrawn from the atmosphere and returned to the environment when a polymer degrades. In comparison to petroleum-based polymers, bio-based polymers have a number of benefits, including being entirely renewable, using less energy during manufacture, being safe (non-toxic), and being ecologically benign [25]. However, compared to resins obtained from petroleum resources, therefore their production costs are 10–12% greater. Some of the advantages of biobased resin are that they are biodegradable in nature, non-toxic in nature, low in cost etc. whereas the disadvantages are that they possess poor impact strength, have low decomposition temperature and brittle in nature.

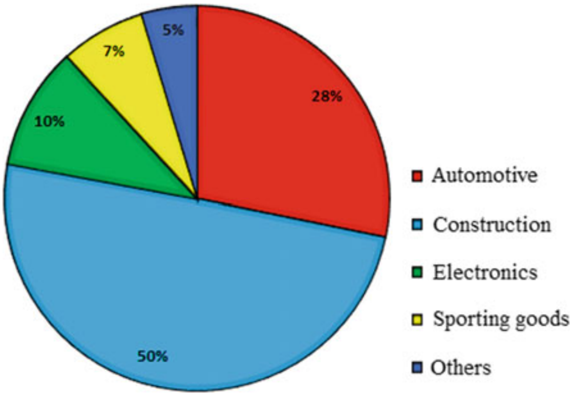
3 Recent Market Trends of NFRPMC

Composites are being used in several sectors for a wide range of products. Numerous nations have taken initiatives and strict environmental rules to encourage the usage of environmentally friendly products and manufacturing practices. The market utilization of NFRPMC in multiple industries is depicted in Fig. 3, along with the range of applications for NFRPMC. Currently, the construction industry is the largest market for NFRPMC. Natural fibre-based composites have always been heavily influenced by the automobile sector. The automobile sector developed natural fibre-based composites and bio-composites in the 1990s, and as a result, their quality and recognition have increased, luring additional industries to the concept that NFRPMC may eventually replace man-made fibre-based composite. Synthetic fibre manufacturing is strongly reliant on petrochemical-based resources, which are also soon running out. These resources also create hazardous waste and by-products, which have an impact on landfill sustainability [41, 42]. Currently, natural fibre-based composites are being utilized in other industries, such as construction. The market has expanded swiftly due to increased manufacturing, and the need for sustainable products has grown while preserving similar qualities. Goods in the construction companies such as siding, decking, flooring and railings started utilising NFRPMC effectively in the market. The automotive sector has the largest influence, even though many other manufacturers and businesses increasingly use more environmentally friendly products. Other businesses have been involved in the manufacturing of NFRPMC by the automobile sector. Natural fibre-based composites are produced using manufacturing techniques similar to those used to create synthetic composites [6, 8].

4 Challenges in Using Natural Fibres as Reinforcement

Natural fibres used in polymeric composites have a lot of drawbacks, including poor mechanical properties, poor interfacial bonding of the fibre and matrix, and moisture absorption. The serious problem of deterioration the NFRPMC experience is when exposed to the outside environment. Degradations caused by biological, mechanical, water, weather, and fire are among them. Due to biological organisms invading NFRPMC, biological deterioration occurs. Hemicelluloses are the primary targets of organisms and enzymes, which hydrolyse them into digestible units. By reducing the fibre-matrix interaction, the strength of composite materials decreases. This reduces

Fig. 3 Global Market consumption of NFRPMC



the strength of composites by weakening the fibre-matrix contact. Enzymes carry oxidation and reduction processes for biodegradation hence toxic chemicals can be avoided by substituting them [43]. The next problem with these composites is water deterioration. NFRPMC absorb moisture in outdoor environments since they are hydrophilic. It is controlled by the number of voids in natural fibres and the amount of non-crystalline components. The main sources of this absorption are rain, salt-water, dew, and ice. Hemicelluloses are the primary component of fibre that absorbs moisture [9]. This causes the fibre to inflate, and the fibre also contracts when it dries. Wetter and drier locations within NFRPMC are observed which are regularly exposed to water that results in differential swelling and eventually cracking. The hydroxyl group on fibres weakens the link between the fibre and matrix, reducing the mechanical strength [44]. The natural fibres' tendency to absorb moisture restricts the outdoor uses of NFRPMC. To decrease moisture absorption and improve mechanical qualities, a variety of techniques, including chemical treatment coupling agents, are employed. It has been shown that fibres and composites' physical and mechanical properties improve with decreasing moisture content [45]. When these composites are exposed to surroundings, ultraviolet (UV) radiation can contribute to degradation. The fibre's lignin component is what causes this weather deterioration. After the initial layer of lignin has degraded, the fresh layer is exposed to UV light. This causes a substantial waste of fibres near the composite's surface. Wind, dust, snowfall, rain, and other factors degrade NFRPMC mechanically, causing stress, fracture, cracking, and erosion. The low fire resistance of NFRPMC is another significant obstacle to their utilisation in diverse structural applications. Additionally, there is very little data known on the function of such composites. NFRPMC are destroyed by fire owing to fast pyrolysis (oxidation, hydrolysis, oxidation). As the temperature increases, such reactions occur. Initially, Hemicelluloses get degraded first, and cellulose, then lignin. The thermal degradation process consists of two steps, the first of which begins between 220–280 °C and the second of which begins between 280–300 °C. Hemicellulose degradation is connected to the lower temperature range, while lignin degradation is tied to the higher temperatures. Natural fibre's colour, smell, and mechanical qualities decline due to thermal decomposition. The degradation processes follow four stages viz, oxidation, reduction, hydrolysis, & dehydration indicating a common degradation chemistry. Another major problem is the uniformity and quality of the fibres used in the composite. The diameter of a fibre varies during its length and is occasionally not exactly round. Low impact strength and flaws in the composite are characteristics of natural fibres. A significant issue in NFRPMC is the poor matrix-fibre interaction. Lower durability caused by fibre degradation that occurs after composite production can be significantly improved by fibre surface treatment [43, 46].

The biggest challenge which is associated with natural fibre-reinforced polymeric matrix composites is due to the existence of surface impurities, water degradation, thermal and fire resistance etc. Although natural fibre-based polymeric composites have many advantages, they also have several disadvantages, such as moisture absorption, subpar mechanical performance, and insufficient interfacial adhesion between the fibre and the matrix [47–49]. Natural fibres lose their mechanical properties when impurities like lignin, hemicellulose, pectin, & wax are present in the natural fibres. The interfacial adhesion between the matrix and the reinforcing component is hampered by these impurities. For instance, the sensitive nature of natural fibres towards temperature and moisture limits their application to the interior parts of automobile vehicles (e.g., door panels, trim in dashboards, parcel shelves, cabin lining, seat cushions, head and backrests). Additionally, the major challenge regarding NFRPMC is water degradation. They absorb moisture from the external atmosphere because of their hydrophilic nature. Hemicellulose is the main component which is accountable for absorbing moisture in the natural fibre. When the fibre dries out, it contracts and when it absorbs moisture, it expands, which finally causes cracks in the composites [50, 51]. Poor mechanical characteristics of the composite arise from the OH- group of water-soaked fibres forming a bond with one another, which weakens the interfacial bond between the matrix and fibre [52]. The degree of moisture present in natural fibres might be determined by measuring moisture regain [53].

The mathematical equations used to measure the thickness swelling of the composite samples (Eq. 1), water absorbency (Eq. 2), and moisture regain capacity (Eq. 3) of the composites are determined as follows; [53].

$$Ts = \frac{Tw - Td}{Td} \tag{1}$$

where Ts refers to thickness swelling, Td for the sample's initial thickness swelling before water immersion, and Tw for the sample's final thickness swelling following immersion.

$$Wa = \frac{Ww - Wd}{Wd} \tag{2}$$

where Wa stands for water absorbency, Wd is the sample's initial weight before immersion in water, and Ww is the sample's weight following immersion in water.

The ratio of the amount of moisture that remains after drying a material under controlled conditions of temperature and humidity associated with the original weight of the material is known as moisture retention. The value of moisture regain must be evaluated to determine the moisture regain, or moisture absorption capacity of natural fibres.

$$\mathrm{MR} = \frac{100\mathrm{MC}}{(100 - MC)} \tag{3}$$

or

$$MR = \frac{A}{B} \times 100\%$$

As a result, Eq. (3) may be written mathematically as follows: where A is the weight of water and B is the dried weight; MC is the percentage of moisture content; MR is moisture regain; NFRPMC' ability to absorb moisture restricts their application to external automotive components. Various chemical treatment procedures are used to improve the physical and mechanical qualities of natural fibres while reducing the amount of water absorption and surface impurities. According to several reports, natural fibre-based composites have greater mechanical characteristics when there is less moisture present in the fibres [53, 54].

5 The Thermal Resistance of NFRPMC

Since NFRPMC have high thermal resistance, using thermal insulating materials is an effective way to save energy costs and increase their industrial effectiveness in fields including construction, packaging, and the automobile industry. Natural fibres include lumens, or hollow pockets full of air. These findings show that the thermal resistance of the NFRPMC falls as the fibre loading increases. As a result, the thermal conductivity of NFRPMC can be improved by applying various chemical treatments to the natural fibre. Alkalization, benzoylation, silane, and acetylation are a few chemical treatments that can improve the thermal insulation and thermal conductivity of composite materials [55, 56].

6 Fire Resistance of NFRPMC

Polymer matrix degrades fast when subjected to high temperature. The thermal stability of composite materials at high temperatures can be enhanced by fibre reinforcement. One of NFRPMC' weaknesses is that at elevated temperatures, they lose their stiffness and strength [57] NFRPMC experience thermal degradation and combustion when exposed to fire. Because of the differences in their morphologies and chemical compositions, different types of natural fibres have varying degrees of flammability. Natural plant fibres begin to degrade at around 220C, while lignin degrades at a temperature of around 200C [58–61]. But whereas cellulose and hemicellulose decay at higher temperatures [62]. NFRPMC' fire resistance properties can be increased by reducing the cellulose percentage of the fibre, enhancing crystallinity, and reducing composite polymerization [57]. Additionally, coatings and additives like silicone, ceramics, intumescent, phenolics, ablation, and glass matting can increase the fire resistance of NFRPMC. A cellular and charred surface is formed when intumescent materials are heated over a particular temperature, protecting the underlying substrate from heat and combustion. By establishing heat barriers, the incorporation of surface treatments, talc & nanoparticles as filler to the NFRPMC can boost the fire resistance properties of the composites [61].

7 Surface Treatment of Natural Fibres

The main drawbacks of employing natural fibres as reinforcement in composites include high moisture retention by these fibres and poor adhesion between the polymer matrix & fibre interface. Natural fibres lose their mechanical properties when non-cellulosic components like lignin, hemicellulose, pectin, wax, etc. are present on the fibre surface which therefore hinders the interfacial bonding between the matrix and the reinforcing material. Therefore, to optimise their bonding with different matrices, natural fibres' surface properties must be modified. To improve bonding between the matrix and the fibre interface, natural fibre surfaces can be enhanced using two methods [63].

- Physical treatment method
- Chemical treatment method.

7.1 Physical Treatment Method

Physical treatment methods modify the surface characteristics of natural fibres without altering their chemical composition which enhances the properties of NFRPMC. Physical treatments include the use of argon treatment, laser treatment, heat, and plasma treatment\. these treatments improve the thermal, mechanical, and physical characteristics of surface-modified natural fibres over chemical treatment methods. Currently, chemical treatment procedures are preferred by scientists over physical treatment methods due to the cost difference between the two [62].

7.2 *Chemical Treatment Methods*

The hydrophilic characteristics of natural fibres and the hydrophobic characteristics of the matrix are the main issues that NFRPMC encounter. As natural fibre & polymer matrix are incompatible, there is poor interfacial bonding between them. By imparting chemical treatments to reinforcing natural fibres, the hydrophilicity may be decreased, improving the interfacial adhesion with the matrix phase [64, 65]. Extensive research has been done to enhance the interfacial bonding between the matrix and reinforcement material by different chemical treatments. The following presents several chemical treatment techniques used to lessen the hydrophilicity of the natural fibre and their implications on composite performance. (i) Alkalization treatment, (ii) silane treatment, (iii) Acetylation, (iv) Maleated coupling, (v) Benzoylation treatment.

- **Modification using Alkalization treatment**

The alkalization treatment method involves treating natural cellulosic fibres with sodium hydroxide (NaOH) solution to eradicate non-cellulosic substances from the surface of the fibre cell wall, such as lignin, hemicellulose, pectin, wax, & oils. When a fibre is reinforced with thermosets and thermoplastics, it changes the cellulosic molecular structure of the natural fibre [66]. The alkalization reaction mechanism on natural fibre is presented in Fig. 4. Treatment with NaOH helps in the elimination of surface impurities along with waxes, lignin, and hemicellulose which makes the surface of the natural plant fibres rough. This surface modification with alkali increases the hydroxyl group's significance for the interaction with the hydrophobic polymer matrix, which strengthens the adhesion or bond between the fibre & matrix [67, 68].

- **Modification using silane treatment**

The hydrophilic property of the fibres is interrelated to their surface energy. Silane, a versatile chemical behaves as a coupling agent which is utilised in the modification of the fibre surface Fig. 5 [69]. The silanes that are often utilised may provide

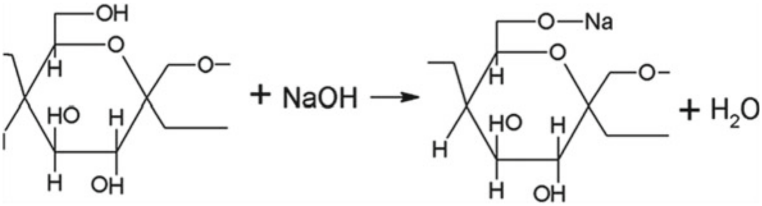


Fig. 4 Reaction mechanism of NaOH on natural cellulosic fibre [63]

$$CH_2CHSi(OC_2H_5)_3 \xrightarrow{H_2O} CH_2CHSi(OH)_3 + 3C_2H_5OH$$

$$CH_2CHSi(OH)_2O + \text{Fibre-OH} \longrightarrow CH_2CHSi(OH)_2O\text{—Fibre}$$

Fig. 5 Reaction mechanism of Silane on natural cellulosic fibre

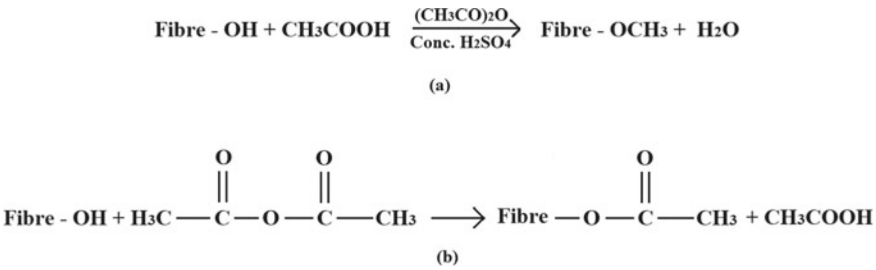
hydrophilic characteristics to the fibre substrate. this treatment includes the application of vinyltrimethoxysilane or aminopropyl triethoxysilane [70]. Natural fibres comprise tiny pores or voids within. Therefore, surface coating of the natural fibres is performed by using silane coupling agents. Due to the coupling agent's ability to enter the micropores or voids, interlocked coatings are generated on the surface of the fibre which decreases the polar component and the surface energy of the natural fibres. In comparison to alkaline-treated NFRPMC, silane-treated NFRPMC exhibits superior mechanical properties [71].

- **Modification using the acetylation process**

Another chemical process for modifying natural fibres is acetylation, which converts the hydroxyl (OH-) group of the cellulose structure into an acetyl group, which increases its hydrophobicity. The primary goal of this chemical process is to encapsulate the OH- groups and impart hydrophobicity to natural fibres. When these hydrophobic fibres are used in fabricating NFRPMC, it hereby improves the overall dimensional stability of the composites [72]. Furthermore, the acetylation process makes the surface of the fibre rougher and leaves fewer spaces, which improves the interfacial bonding with the matrix. In this procedure, the cellulose structure of the fibre is grafted by an acetyl group both with and without the presence of a catalyst (Refer Fig. 6). Fibres often don't react well with acetic acid or acetic anhydride. To lessen the hydrophilic properties of natural cellulose fibre, a significant of acetylation is used. Additionally, it was discovered that natural fibre-reinforced polymeric composites had improved flexural and tensile characteristics. Also, acetylation enhanced matrix interphase & fibre adhesion. Along with better surface morphology and stress transmission from the matrix to the fibres, the composite also achieves improved tensile strength [73, 74].

- **Modification by using a maleated coupling agent**

The maleated coupling technique is frequently utilized to impart hydrophobic character to the natural fibres. Maleated anhydride is mostly used to modify the fibre surface to enhance the interfacial adhesion between the fibre and matrix phase. This enhances the overall mechanical performance of the composites. Maleated anhydride eliminates the OH- groups from its amorphous region to make the cellulose structure compatible with the polymeric resin. This procedure coats the fibre's surface by decreasing the hydrophilic property. The OH groups of the fibre and the anhydride groups of the maleic agent form a carbon–carbon covalent bond due to the maleated coupling agent. This covalent bond creates a bridge junction that strengthens the



$$\text{Fibre - OH} + CH_3COOH \xrightarrow[\text{Conc. } H_2SO_4]{(CH_3CO)_2O} \text{Fibre - O}CH_3 + H_2O$$

(a)

$$\text{Fibre - OH} + H_3C-\overset{\overset{\displaystyle O}{\|}}{C}-O-\overset{\overset{\displaystyle O}{\|}}{C}-CH_3 \longrightarrow \text{Fibre}-O-\overset{\overset{\displaystyle O}{\|}}{C}-CH_3 + CH_3COOH$$

(b)

Fig. 6 **a** Acetylation reaction by using an acid catalyst **b** Acetylation reaction without using an acid catalyst

bonding between the polymeric matrix and the fibre. The polypropylene chain renders the maleic anhydride coupler cohesive by treating cellulose fibres with hot MAPP (Maleic anhydride-grafted polypropylene) copolymers, which creates a covalent bond formation at the surface. The esterification process of the cellulose structure of the fibre and the activation energy of the copolymer when heated is explained by the reaction between MAPP & cellulose structure of the fibre is exhibited in Fig. 7. The hydrophobicity and bonding at the surface interface between the fibre and polymeric matrix are enhanced by this method of fibre surface treatment. Additionally, the treated fibre's hydrophilic tendency was diminished. Additionally, NFRPMC greatly improves hardness, flexural modulus, young's modulus, & impact strength compared to those of untreated fibre composites [75].

- **Benzoylation treatment**

To reduce the amount of moisture from the natural fibre cell wall, the benzoylation treatment method is carried out by using a benzoyl chloride solution. This enhances the bonding between the fibre & matrix interface, which increases the strength of the composite, and improves the natural fibre's thermal stability [76]. The fibres utilised for this procedure have already been alkali-treated. The Benzoylation process helps in removing hemicellulose, lignin, waxes, & oil substances from the cellulosic fibre structures, which leaves a significant number of OH- groups exposed on the surface of natural fibres. In this procedure, the natural fibre's OH- the group gets replaced by a benzoyl group, which bonds with the fibre's cellulose structure (illustrated in Fig. 8) [77, 78]. Therefore, the benzoylation process reduces the treated fibre's propensity to be hydrophilic and further improves the interfacial interaction with the hydrophobic matrix. Also, when compared to untreated fibre-reinforced composites, benzoyl chloride treatment enables the fibre composites to be more thermally stable [78].

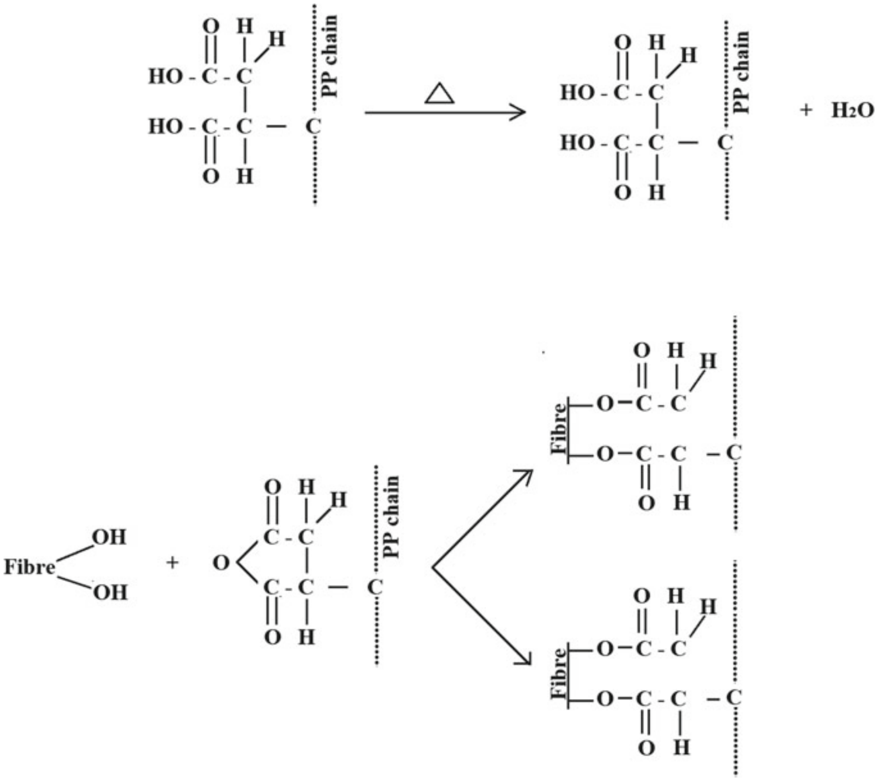


Fig. 7 The reaction mechanism of cellulose fibre with MAPP copolymer [75]

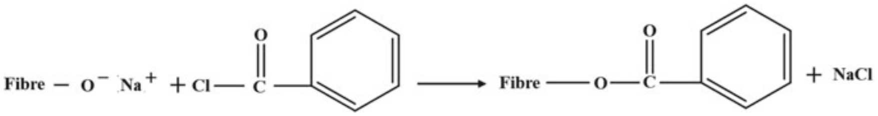


Fig. 8 Reaction mechanism of natural Fibre with Benzoyl chloride

8 Future Challenges of NFRPMC & Their Possible Solutions

In various application areas, NFRPMC have had tremendous success in research and development along with commercial success. The below Fig. 9, presents a SWOT analysis of natural fibres as a technologically advanced raw material. SWOT stands for Strength, Weakness, Opportunity, & Threat. This is not a comprehensive list of SWOT. The elements in the strength, as well as opportunity segments, are anticipated to increase in the future, while the elements in the weakness & threat segments

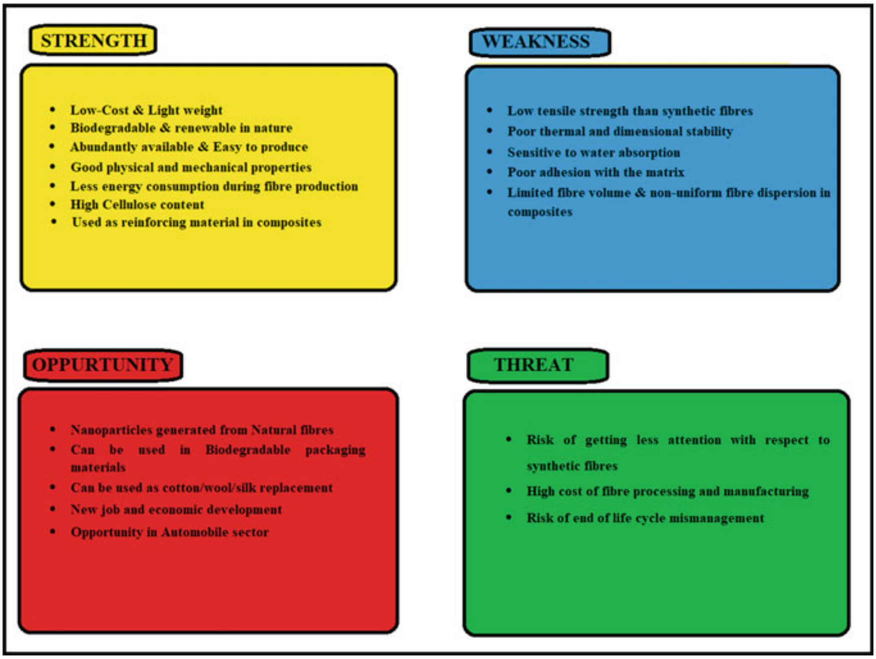


Fig. 9 SWOT analysis of natural fibres

are expected to diminish. Some of the significant problems with natural fibres are discussed in this section [79].

There is a need for more intensive research to be done on the durability, safety and recyclability of NFRPMC. Several safety concerns arise during the recycling of formaldehyde-based adhesives such as the release of volatile organic compounds. Composites are found to be less durable when they are used for exterior application as they are susceptible to extreme weather conditions, microbial attacks, etc. NFRPMC are recyclable. But their recycling and applications have several challenges such as their high moisture content, poor bonding between hydrophilic fibres and hydrophobic polymers, flammability, the inconsistent chemical composition of natural fibres, etc. It is essential to identify the process of enhancing the properties of NFRPMC for specific end-use applications before and after recycling for their increased utilisation. To improve the mechanical properties and interfacial adhesion of NFRPMC fundamental research must be carried out on the fibre matrix interface [80].

To enhance the properties of composites some physicochemical techniques have been suggested such as mechanical hot-pressing along with the usage of acids or epoxy as modifiers. Graphene-based materials are used in an emerging class of natural fibre composites to provide reinforcement or to improve the interfacial bonding between the hydrophobic polymeric matrix & hydrophilic natural fibres. The toughness and stiffness of NFRPMC are observed to enhance with the addition of graphene-based materials. Broader application of NFRPMC and their recyclates require more investigation of durability, cost reduction and sustainability. With the help of newly developed mitigation techniques used for recycling, composites can achieve long-term durability and recyclability along with high performance. Composites comprised of polymers and wood fibres are known as wood-plastic composites. Wood is necessary for sustainable economic development, but the thermoplastics obtained from fossil fuels have negative environmental impacts throughout the composite's life cycle. After use, wood plastic composites are primarily treated as waste to be burned, which results in value loss and environmental problems. An eco-friendly acceptable way is to recycle wood plastic composites. Recycled plastic-based wood plastic composites are preferred to those made of virgin plastic. Alternatives that are both more advanced technologically and environmentally friendly are wood-plastic composites produced from recycled polymer. However, additional separation and particle processing may be required for preparing recycled composites which harm the environment [81]. For instance, the durability of composites is improved by adding additives. But adding additives has negative effects on human health and the environment throughout their life cycle. Additionally, novel high-performance biobased polymers derived from biomass can be used to improve the sustainability of such composites. The cost of the composite can be minimised by using cost-effective methods and the reuse of raw materials such as polymers, waste fibres, etc. It would be very helpful to the field if the further study were done on the influence of processing variables on the characteristics of recycled NFRPMC. NFRPMC are often prepared and recycled using processing techniques such as mixing, compounding, extrusion [82], compression moulding and injection moulding, these variables have a substantial impact on the characteristics of all polymer composites. For various processing techniques, the parameters that can be changed can also vary. Wide-ranging uses of thermoset composites suggest that there is a potential market for recycling used thermoset composites. However, there is limited research done on the recycling of thermoset composites reinforced with natural fibres produced bio-composites using humans-based thermoset resin, chicken feathers and vegetal non-woven fibres (linseed flax and jute). Further, they have performed mechanical recycling of resin and composites [15]. They reported that when exposed to heat, humans-based resin undergoes structural and chemical changes that lead it to become flexible. This resin is suitable for mechanical recycling as it can be reprocessed under pressure and heat into the desired shape. It was observed that the thermal stability of the composites was not drastically affected after recycling also surface morphology of recycled composites revealed that the dispersion of fibres was non-directional but consistent. Additionally, it was noted that there was inadequate fibre-matrix adhesion as well as holes and cracks on the fibre-matrix interface. Recycling thermosets is still difficult because of the strong chemical cross-linking in cured thermosets. Thermosetting composites will require cost-effective recycling. Adulteration and immiscibility of polymers during recycling is a major obstacle to the external recycling of NFRPMC. It is possible to build efficient physical pre-sorting of the mixture's constituent parts (for instance, sorting by size, weight, or colour) [83].

8.1 Inferior Properties of Natural Fibre

Natural fibres, despite having several positive properties, face several obstacles to becoming a preferred biobased sustainable fibre on the worldwide market. However, several issues need to be marked, such as the fact that natural fibres are less strong than synthetic fibres, that they degrade thermally at low temperatures, that they are more susceptible to water absorption and that there are variations in the performance and quality of the fibres. The functional properties of natural fibres can be enhanced through fibre modifications using several inventive chemical or physical treatments, such as treating fibres with various nano-particles [84].

8.2 Challenges in Composite Processing

The reliability of the quality of natural fibre-based composite products using conventional composite production methods continues to be a significant problem. To produce high-quality, consistent products with high fibre content, new composite manufacturing methods should be evolved with a focus on optimizing the processing parameters. One method is to use fibre instead of the more common matrix types like granule, sheet, or plate, which can increase the composite's fibre volume ratio. The development of new methodologies is required to assure composite quality. The processing costs or the cost per product unit must be kept to a minimum while the quality of the products is being improved. There have been some attempts to print natural fibre composite using the 3D printing process, however void formation and low fibre content issues still need to be fixed [84].

8.3 Issues with Composite Performance

Some inherent faults of Natural fibres are carried over into their composite forms as well. Still, natural fibre composites are weaker than some natural fibre composites and popular synthetic fibre composites. Poor matrix bonding is thought to be the cause of the composite's problems. Additionally, the composite cannot produce lightweight, strong and environmentally friendly composites due to the limited fibre volume (20% to 40%). Hybridization and fibre surface modification can assist to enhance the characteristics of the composites by encouraging a strong bonding of the fibre, which is necessary to compete with the other high-performing synthetic-fibre-reinforced polymer composites. Natural fibres and polymers can get easily bonded when treated with nanoparticles, allowing for higher fibre volumes in composites. A few biobased and biodegradable polymer matrixes are now on the market. It is necessary to conduct additional research to create a novel-based polymer matrix. The chemicals used to treat fibres may harm the environment. Therefore, it is necessary to conduct research to identify beneficial natural biobased extracts and natural dyes for treating fibres.

A mixture of natural fibres treated with biobased chemicals or other biodegradable chemicals and biobased and biodegradable polymer matrix with organic fibres [85]. Natural fibres treated with bio-based chemicals or other ecologically sustainable, but a lesser amount of energy-consuming physical procedures can be used to create a truly green composite. Water absorption by the composite could result in natural fibre swelling, which eventually compromises performance by causing the fibres to separate from the matrix interface. This problem might be reduced by new waterproof coating technologies. Studies evaluating the mechanical performance of composites following thermal or physical ageing or long-term performances like creep behaviour are still insufficient [86].

8.4 Lack of Product Diversification

Due to insufficient global marketing efforts and a lack of product variety, the number of composite products containing natural fibres and their market share is still relatively low. It is necessary to develop novel concepts with applications in mind for both high-value and widely consumed goods. Although there are several novel concepts for natural fibre-based goods that have generated a lot of research attention, such as cotton replacement and nanocomposite preparation, there is still a need to transmit the research's findings to the market for commercialization. The items made from natural fibres must be promoted extensively to attract buyers and traders from around the world, such as by exhibiting at international trade shows. This will provide opportunities for new research initiatives with international partners and inspire foreign businesses to engage in the development of products manufactured from natural fibres. Countries like India, Bangladesh and China, which are top exporters of natural fibres should collaborate on research to come up with fresh concepts to encourage the diversification of natural fibre goods on global markets. Its extensive use in domestic and international markets can be facilitated by coordinated policy development by the nations that produce natural fibres. Technological advances in fibre and composite processing are expected to encourage quality enhancement and the expansion of the market for a variety of natural fibre products [86].

9 Recycling, Biodegradability and Circular Economy Issues

Despite the significant financial and environmental advantages, the industry is very concerned about the end-of-life cycle of natural fibre-reinforced composites due to improper management. This is perhaps because many plastics, particularly thermoset plastics, which are difficult to recycle, are burned or dumped in landfills. Even if it is recycled, the cost of recycling is. can still outweigh the value of the material recovered. As a result, creative recycling methods or tactics must be created.

Natural fibre composites made of biopolymers are degraded biologically rather than being recycled, reused, or remanufactured to increase their lifespan. Special consideration must be given to this area for future study. The circular economy strategy promotes resource recovery from waste economically. The circular economy system should be used to manage the end life of Natural fibre reinforced composites through efficient material recovery and the application of innovative technologies. The recycling of synthetic and natural fibres from the carpet industry showed some encouraging outcomes [86]. Despite the widespread adoption of synthetic fibre recycling practices, natural fibre thermoplastic composite recycling does not receive the attention it deserves because it is produced at a far lower scale than synthetic fibre thermoplastic composites. 352,000 tonnes, or around 15% of global production of fibre-reinforced composites, are made up of biocomposites (natural fibre and wood polymer composites). The ecosystem is not harmed by natural fibres either. Their safe deterioration, low cost and lower energy burden during production are the reasons they are chosen (low carbon footprint). The market for NFRPMC is anticipated to grow significantly, reaching USD 6.5 billion in 2021. As a result, finding a trustworthy way to recycle this kind of engineering NFRPMC is now a crucial issue. To implement the circular economy paradigm for bio-based composites, the life cycle framework must be taken into consideration [87, 88]. At each stage of the product life cycle, assessing the ecological consequences and life cycle costs will help in making decisions for sustainable product development [89].

9.1 Recycling of Natural Fibre-Reinforced Composites

Polymer composites that are loaded with particles or strengthened with fibres make recycling more challenging. The aim is to segregate the material to be recycled for reuse in the same application from the fibres or fillers if the polymer is no longer being used.

- **Mechanical recycling**

The mechanical approach aids in obtaining fillers or granulates prepared for additional thermal shear procedures like extrusion and injection moulding (in the case of thermoplastic composites). It is a common practice to add virgin raw materials to the recycling process. It is worth noting here that mechanical recycling has only an effect on the long fibre's length. This means that for short fibres composites, no difference is to be expected [87].

- **Chemical recycling**

Chemical recycling that uses the solvolysis process is categorised as low temperature or supercritical temperature or at temperatures more than 400 °C. The solvolysis process was created primarily to remove the expensive carbon fibres from the epoxy matrix of carbon fibre composites, which have several restrictions on landfilling. Since the entire matrix, including Polyester, Polycarbonates, nylon, and Polyurethane, must be dissolved using chemicals that are harmful to the environment, the procedure is both exceedingly expensive and environmentally damaging. Solvolysis is a depolymerization method that breaks C-X bonds to generate oligomers or monomers, much as alcoholysis, hydrolysis, acidolysis, and aminolysis. Where X stands for a heteroatom, including halogens, Oxygen, Nitrogen, Phosphorous, and Sulphur. Thermal procedures like the fluidized bed process and pyrolysis rely on burning the matrix to recover the fibres. The most common recycling method, which yields granules or pellets for injection, is re-melting and remoulding because the thermoplastic matrix is thought to be an easily recyclable matrix. This method is also preferred over chemical and thermal recycling. Since it protects the fibre from severe thermal or chemical degradation, especially when reinforced with natural fibres, this approach is often advised. The primary and secondary mechanical recycling processes are particularly suitable for the reuse of thermoplastic composites which are reinforced with synthetic fibres. By reducing the molecular weight during the subsequent mechanical recycling, the host matrix may undergo some kind of thermal degradation. However, it is not anticipated that the synthetic fibres will be impacted by heat. On the other hand, it is anticipated that natural fibres will break down due to temperature action. Similarly, recycling operations have an impact on the length of the synthetic fibre. It is believed that the initial fibre size and composition had an impact on this. In synthetic fibres, shortening occurs through fibre fragmentation, but in natural fibres, it is thought to occur in additional ways, such as fibre splicing. Additionally, the thermal breakdown or volatilization of some fibre components may result in a reduction in fibre size [88, 89].

10 Conclusion

The increasing future demands to create biodegradable natural fibre reinforced polymeric matrix composites have been sparked by the rising needs for renewable & recyclable materials, worrying energy catastrophes, environmental legislation, and lastly due to plastic waste & pollution. There is a promising substitute for these recyclable and unsustainable synthetic fibres and polymers in the shape of natural fibres. Natural fibre-reinforced composites promise to have a bright future for a variety of applications. These composite materials, which have a variety of physicochemical & mechanical properties, might be good enough to displace the current range of synthetic materials which are derived from fossil fuels. Further, future challenges and difficulties in manufacturing natural fibre-reinforced composites are being researched due to the abundant availability of natural fibres, along with their distinctive properties, and environmentally friendly behaviour when compared to synthetic polymers and fibres. However, to effectively compete in the global composites industry, a paradigm change in the manufacture of composites and cost reduction would be required for future advancements in natural fibre-reinforced composites.

These composites will eventually exhibit better mechanical and functional properties which are equivalent to those of synthetic composites. The following are the anticipated future development trends for natural fibre-reinforced composites: first, low-cost manufacture which will receive overall acceptance. Secondly, prices will considerably drop due to the increased market demands, increased production of inexpensive natural fibres & polymers, and due to mass production of natural fibre composites. Therefore, ongoing and future research should focus on fabricating and improving NFRPMC with various natural fibres, ratios, and shapes for multifunctional applications. Due to the complex and varied character of natural fibres, an appropriate database of fibres and composites should be generated. Composites that are completely biodegradable and have excellent multifunctional properties will soon be a reality. Fibre surface properties and the fibre/matrix interface may be improved through fibre modification methods, such as surface treatments with chemicals like silane, alkali, coupling agents, etc. This may lead to superior NFRPMC that can satisfy several requirements and are on par with composites based on synthetic fibres. In addition, research into the tribological characteristics of natural fibre composites will be enhanced due to the integration of nanocellulose and/or nano clay into composites, which will further enhance numerous physicochemical properties, which would be of key interest in terms of future challenges and difficulties.

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Effect of Interfacial Bonding Characteristics of Chemically Treated of Various Natural Fibers Reinforced Polymeric Matrix Composites

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Abstract Bio-composite materials, which are formed of natural fibers and a polymer matrix, represent an engineering composite product that can be beneficial for a diverse range of uses. These materials are being used in an ever-expanding variety of applications because of the extraordinary qualities they possess, the varied designs they come in, and the appealing ways they may be put to use. Nevertheless, the application of these bio-composites is reliant on the interface bonding between fiber and matrix internal bonding in order to achieve the desired level of performance. The interfacial connection that exists between the fiber and the matrix has a considerable impact on the physicomechanical characteristics of the bio-composites. Numerous researchers have been motivated to investigate natural fibers' potential applications in a broad selection of industrial sectors as a consequence of the fact that natural fibers are readily accessible, inexpensive, and biodegradable. There are several drawbacks associated with these fibers, including the fact that they have an extreme moisture absorption degree, which causes to an increase in the thickness swelling, they are simple to degrade, they have a low resistance to fire, they have heterogeneity in their mechanical characteristics and that they have poor interface bonding with the polymer matrix. With the intention of enhance the bonding among fiber-matrix adhesion and, by extension the physicomechanical properties of the composites, a number of chemical modifications are implemented. This chapter offers a comprehensive review of the chemical treatments and uses for a broad range of natural fiber-reinforced composites that are currently available.

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1 Introduction

A bio-composite is a substance that is consist of two or more diverse constituent materials, at least one of which is derived from a natural source. These constituent materials are mixed to create a material that is superior to the constituent materials individually, and this material is known as a bio-composite. Bio-composite materials are becoming increasingly popular as a result of the fact that they are both environmentally friendly and biodegradable. It can be found in a wide selection of forms, such as films, membranes, moldings, coatings, particles, fibers, and foams, among others. Bio-composites, in contrast to synthetic materials, are capable of decomposing without the release of harmful gases or toxins. This type of composite gets thrown away and converted into mulch without having any negative impact on the surrounding environment. Because of the ecologically favorable properties that they possess, bio-composite materials are finding increasing use in industry. According to Amiandamhen et al. [1] the primary source of strength in bio-composite comes from the fibers, whilst the matrix is responsible for transferring tensions between reinforcing fibers while simultaneously holding the fibers together.

During the creation of composites, natural plant fibers are frequently employed both as reinforcements and as fillers. It is a sort of renewable resource that can be utilized in the creation of novel reinforcements and additives for composite materials that are based on polymers. They can be broken down further into numerous groups based on their biological sources, for instance, bast fiber, leaf fiber, seed fiber, grass fiber, and wood fiber (Fig. 1) [2–4]. The rigidity, flexibility, specific intensity, and stiffness of natural fibers are all significantly higher unlike those of synthetic fibers [5, 6]. As a consequence of this, it is possible that the markets will show a greater interest in this type of fiber. Natural fibers are a capable material that can replace synthetic materials and the goods that are linked with those synthetic materials in applications that require less weight and energy savings.

Natural fibers provide a number of beneficial properties, including superior specific strength and modulus, endurance to corrosion and fatigue, low cost, and low self-weight [3, 7–9]. Processing flexibility, high specific strength, and low self-weight are further benefits. In contrast, it has a numerous of downsides, including a high water absorption rate, a steep anisotropy, poor compatibility with traditional resins, and a poorer homogeneity than glass and carbon fibers [10]. Polarity is a limitation of natural fibers, and the fact that it makes them incompatible with hydrophobic matrices is one of those drawbacks. Because of this incompatibility, the interfacial attachement that exists among the fibers and the matrix is quite low. Directly as a result of this, the mechanical characteristics of the composites suffer [1, 7]. It is possible to work around this problem by chemically modifying the fibers such that they become less hydrophilic.

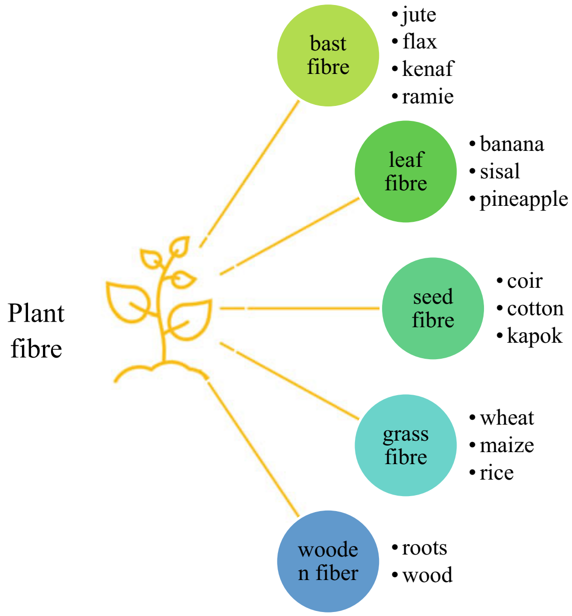

Fig. 1 Five specific types of plant natural fibers

2 Characteristics of Natural Fibers

It is vital to have a thorough understanding of the characteristics of natural fibers to be able to determine the applications that are the most suitable for them, such as bio-composite raw material. Size, shape, crystallite content, orientation, and cell wall thickness are the factors that determine the properties of a single fiber [11]. To be able to get the most out of natural fibers and make them more environmentally friendly, it is essential to take into account their morphological, mechanical, chemical, and biological features. Both tensile strength and density are strongly correlated with the chemical features of natural fibers, such as the presence of cellulose in the cell wall [10]. A significant portion of the cell walls of dried plant fibers, which also contain residues of starch, extractives, protein, and inorganics, is made up of lignin, which is a sugar-based polymer, and sugar-based polymers. Cellulosic fibers are hydrophilic under normal circumstances (absorb moisture). It is possible that the amount of moisture present in the fibers will have a detrimental impact upon the mechanical properties of composites manufactured from natural fibers.

Plant fibers are almost rarely found to exist as individual cells; rather, they are often found to be packed together in groups. In addition, the character of a single fiber is concerned by the structure, size, orientation, and thickness of the cell wall [4, 7]. It is vital to have a thorough understanding of the physical and mechanical characteristics of natural fiber to be able to make optimal use of it as shown in Table 1. In addition to this, the calculation of the weight interaction requires the utilization of a specific quantity. Because of their particular tensile strength and stiffness properties, natural

Table 1 Physical–mechanical properties of selected fibers

Fiber type	Density (g/cm^3)	Tensile strength (MPa)	Elongation (%)	Specific modulus (GPa)	Toughness (MPa)	Latest references
Bamboo	0.91	262	2.7	10.77	–	[12, 13]
Coir	0.87–1.2	95–593	4–47	4.2	10.7	[14, 15]
Kenaf	1.31–1.5	930–1500	1.6–17.3	19.2–36.5	52	[16]
Pineapple	1.32–1.54	413–1627	0.8–27	20.55	95	[17]
Sisal	0.76	274–855	2–7	6.3–12.1	10	[18, 19]
Flax	1.5	1500	3.2	–	–	[20, 21]
Hemp	1.47	690	4	–	–	[22]

fibres are ideally suited for use as reinforcement materials in composites [5]. It is vital to take into account any changes in the quality of the fiber in order to preserve the flawlessness of items created from natural fibers. In addition, it is essential to have a solid understanding of these properties in order to select the most appropriate technique for the cooking process.

To get the most benefit possible from the plentiful supply of natural fibers, it is necessary to conduct research and put those findings to use. The sectors that utilize natural fibres as raw materials have created a significant amount of money for their respective businesses. It is essential that ongoing development take place in order to foster economic expansion and develop the quality of life for communities all across the globe. It might be possible to improve matrix bonding by modifying or pretreating natural fibers in some way. This would allow one to overcome the drawbacks of using natural fiber materials.

3 Natural Fiber Modifications

The most significant drawbacks associated with natural fibers are their high capacity for water absorption and poor dimensional stability. The main aim of fiber modification applied to natural fibres is to enhance the bonding capability and stress transferability of composite materials. These improvements can be achieved through the application of natural fibers. As a whole mechanical characteristics of natural fiber-reinforced polymer composites are greatly impacted by the shapes, quality ratios, hydrophilicity, and structural stabilities of the composites [7, 9, 10]. These techniques usually involve the deployment of chemical functional groups that are adept of interacting with fiber formations in order to alter the structures of those fibers. As an immediate outcome of this, the capacity of the fibers to take up moisture is reduced, which strengthens the suitability of the fibres with the polymer matrix.

Chemically modifying natural fibers results in an enhancement in the adhesion that exists between the matrix and the natural fibers. Wide-ranging study has remained conducted on the topic of the effects of chemical modification on natural fibers [5, 10, 16]. The inherent hydrophilicity of natural fibers can be lowered through the use of a chemical modifying, which also improves the matrix and fiber adhesion mechanism of the natural fibers. Alkaline, acetylation, silane, peroxide, benzoylation, sodium chlorite, maleated coupling agents, isocynate, acrylation and acrylonitrile grafting, permanganate, stearic acid, oleoyl chloride, triazine, and fungal treatments are some examples of chemical treatment procedures [2, 10]. This section will investigate a recent chemical modification of natural fiber that acquire a clearer knowledge of the performance of biocomposites. This section's goal is to focus on the growing necessity for eco-friendly resources in a variety of uses, which is the reason this modification will be explored.

3.1 Alkaline Treatment

Sodium hydroxide (NaOH) is generally utilized to break down the cellulose in natural fibre. The cellulose's crystalline zone collapses as a result, and an amorphous region develops in its place. The addition of NaOH then reacts with the fiber. Due to the interactions, the hydroxyl groups of the molecules are rendered inactive. Subsequently, the hydrophilic characteristic of the fiber eventually transforms into a hydrophobic [23]. The tensile strength of the bio-composite exhibits an improvement as a result of this treatment [24]. Simultaneously, the fibre will be degraded if the alkali proportion surpasses the optimum amount. The fibre's chemical interaction with NaOH is represented by Eq. 1.

$$\text{Fiber} - \text{cell} - \text{OH} + \text{NaOH} \rightarrow \text{Fibercell} - \text{O} - \text{Na} + \text{H}_2\text{O} + \text{impurities} \tag{1}$$

3.2 Silane Treatment

Silane, represented by the chemical formula SiH_4, is an organic compound. Silanes serve as coupling agents, enabling the attachment and stabilization of natural fibers to a polymer matrix. In the occurrence of water, the hydrolyzable alkoxy unit creates silanols, which then catalyse with the fiber's hydroxyl unit to create a powerful covalent bond on the cell wall. Due to the covalent bond, the silane-formed hydrocarbon chains resist fiber swelling by forming a cross-linked network [25]. The reaction is depicted by Eq. 2.

$$\begin{aligned}&\text{CH}_2\text{CHSi(OC}_2\text{H}_5)_3 \xrightarrow{\text{H}_2\text{O}} \text{CH}_2\text{CHSi(OH)}_3 + 3\text{C}_2\text{H}_5\text{OH}\\&\text{CH}_2\text{CHSi(OH)}_3\text{Fiber} - \text{OH} \rightarrow \text{CH}_2\text{CHSi(OH)}_2\text{O} - \text{Fiber} + \text{H}_2\text{O}\end{aligned} \tag{2}$$

3.3 Acetylation Treatment

Acid catalysts are employed in the acetylation process to attach acetyl units to the fibers' cellular formation. Since the fibers cannot be significantly changed by acetic acid or acetic anhydride on their own, the reaction must be sped up by first immersing the fibers in acetic acid, then subjecting them to an acetic anhydride treatment for one to three hours at high temperatures. This process is repeated until the desired result is achieved. This process involves OH and the carboxylic/anhydride groups of natural fibre undergoing an esterification reaction. During the acetylation process, the cuticle and wax that were previously present on the surface of the fiber are eliminated, resulting in a smoother surface [26].

3.4 Benzoylation Treatment

The most common application for benzoyl chloride is the treatment of fibers. Because of the presence of benzoyl ($C_6H_5C = O$) in benzoyl chloride, the treated fiber's hydrophilicity has decreased and its contact with the hydrophobic matrix has improved. Fiber benzoylation creates fiber-matrix adhesion, which greatly improves heat stability, reduces water absorption, and increases composite density [27]. The reaction is depicted in Eq. 3.

$$\text{Fiber} - \text{O} - \text{Na}^+ + \text{CIC} = \text{O} - \text{C}_6\text{H}_6 \rightarrow \text{Fiber} - \text{O} - \text{C} = \text{O} - \text{C}_6\text{H}_6 + \text{NaCI} \tag{3}$$

3.5 Peroxide Treatment

The treatment with peroxide induces peroxide-induced polyethylene to bond to the outside of the fiber and interacts with the OH units that are present in both the matrix and the fiber. Using this approach will expand the linkage of the fibers to the matrix at the boundary, lowers the amount of water that is absorbed and boosts the thermal steadiness of the fibers [28].

3.6 Isocyanate Treatment

The strength of the bonding chain can be improved with an isocyanate treatment. This is accomplished through the establishment of stronger covalent interactions between the matrix and the fibers. In addition to this, it makes the fibers further resistant to moisture. The consequence of interaction between isocyanate functional groups (-N = C = O) with the OH units of the constituents of lignin and cellulose results in the formation of strong covalent connections (chemical bonding) and resistance to moisture [29].

3.7 Maleated Coupling Agent

Maleated coupling agents have the ability to establish carbon–carbon bonds with the polymer chains that make up the matrix, allowing for improved interaction with the matrix's functional surfaces as well as the fibres. As a result of the reaction between the hydroxyl group in the amorphous region of the cellulose structure and the maleic anhydride, the surfaces of the fibers are coated with polymers that are formed of long-chain molecules. As a consequence of the formation of covalent bonds between the OH groups of the fibers and the anhydride groups of the maleic anhydride, the coating is able to remove the OH groups after the fiber cells have been formed. This reduces the tendency of the fibers to attract water, and it moves the process along toward establishing a connecting interface and achieving efficient interlocking between the matrix and the fiber [30].

3.8 Sodium Chlorite Treatment

The fibers are bleached by the sodium chlorite treatment by adding sodium chlorite to an acid mixture that already contains sodium chloride ($NaClO_2$). This technique removes any moisture that may be present in the fiber, which in turn increases its hydrophobicity and, consequently, its deformability [31].

4 Interfacial Bonding Characteristics of Chemically Treated Various Natural Fibers-Reinforced Polymer Matrix Composites

When it comes to determining the overall properties of natural fiber-reinforced polymer matrix composite, the effectiveness of the fiber-matrix interface bonding is among the most critical aspects to consider. Lignin, cellulose, and hemicellulose are the three elements that make up natural fibers. The several well-known natural fibers are broken down into their component chemicals and shown with their structural characteristics in Table 2. As can be noted in Table 2, the various natural fibers contain dramatically varying amounts of chemical components. This disparity may be attributable to a number of factors, including place of origin, age, method of

Table 2 Chemical composition of some natural fibers [32, 34]

Types of fibre	Cellulose	Lignin	Hemicellulose	Pectin	Wax	Moisture content
Bast						
Jute	61–71.5	12–13	13.6–20.4	0.2	0.5	12.6
Flax	71	2.2	18.6–20.6	2.3	1.7	10.0
Hemp	70.2–74.4	3.7–5.7	17.9–22.4	0.9	0.8	10.8
Ramie	68.6–76.2	0.6–0.7	13.1–16.7	1.9	0.3	8.0
Kenaf	31–39	15–19	21.5	–	–	–
Leaf						
Sisal	67–78	8.0–11.0	10.0–14.2	10.0	2.0	11.0
PALF	70–82	5–12	–	–	–	11.8
Henequen	77.6	13.1	4–8	–	–	–
Seed						
Cotton	82.7	–	5.7	–	0.6	–
Fruit						
Coir	36–43	41–45	0.15–0.25	3–4	–	8.0

retting, etc. From all the natural fiber that were discussed, coir has the smallest cellulose content and the maximum lignin content relative to its overall composition. Cellulose is a hydrophilic glucan polymer that is fabricated of a linear chain of 1,4-β-bonded anhydroglucose units and an alcohol hydroxyl. Cellulose is also known as microcrystalline cellulose. As seen in Fig. 2, these hydroxyl units make hydrogen bonds both intramolecularly within the macromolecule including intermolecularly with other cellulose macromolecules and hydroxyl groups. Therefore, natural fibers are hydrophilic, and their moisture content ranges from 8 to 12.6% [32, 33].

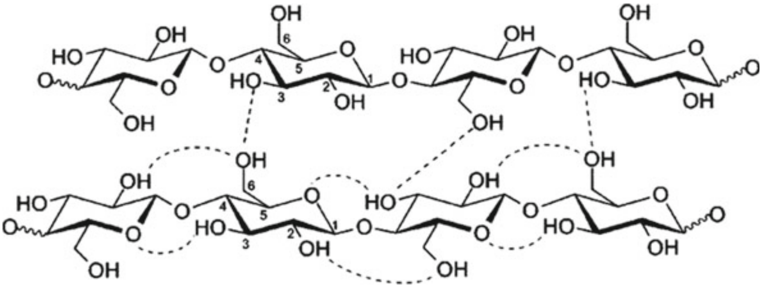

Fig. 2 Illustration of hydrogen bonding in natural fiber [35]

4.1 Kenaf Fiber-Reinforced Composite

During the manufacturing kenaf fiber-reinforced composite, the fundamental constraint that is encountered is an insufficient level of fiber-matrix bonding between the kenaf fiber and the polymer matrix. Accordingly, the composite performance will decrease. This is due to the hydrogen linkage on the the surface of fiber having a tendency to restrict the wetting of the filler surface and polar hydroxyl groups on the surface of the kenaf fiber having difficulty establishing a well-bonded interphase with a relatively nonpolar matrix. In addition, the incorporation of kenaf fiber into a polymer matrix for the purpose of acting as a filler is usually followed by agglomeration. This is because the fibers have a tendency to form hydrogen bonds with one another, which leads to an insufficient level of dispersion. Numerous selection of chemical modification that can be applied to the natural fiber that will improve its adherence to the matrix [36].

The alkaline treatment, also identified as mercerization, is one of the very old chemical treatments for natural fibers [37–40]. This treatment is particularly common for kenaf fiber, which is used to strengthen thermoplastics and thermosets. The fibers are given this treatment by being soaked in a NaOH solution for a considerable amount of time [40–43]. The most noticeable effect of alkaline treatment is the splitting of hydrogen bonds within the system structure, which findings in an increase in the surface's roughness. This process depolymerizes the cellulose, removes the hemicelluloses, lignin, wax, and oils from the exterior surface of the fiber cell wall, and exposes the short-length crystallites. The treatment with alkaline causes the fiber bundles to fragment into smaller fibers, which is known as fibrillation. To put it another way, because of this treatment, the fiber diameter is decreased, which results in an increased aspect ratio. The process is depicted in Fig. 3, which illustrates it. The accumulation of aqueous sodium hydroxide (NaOH) to the kenaf fiber helps to enhance the ionization of the hydroxyl unit to the alkoxide as in Eq. 4 [40, 43].

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber ONa}^{+} + \text{H}_2\text{O} \tag{4}$$

It has been stated that an alkaline treatment has two possessions on the kenaf fiber: (1) it improves surface unevenness, which ultimately leads to improved mechanical interlocking, and (2) it intensifications the amount of cellulose visible on the fiber surface, which ultimately fallouts in growth the amount of potential reaction sites. As a consequence of this, the treatment of natural fibers with alkaline solutions has a long-lasting influence on their mechanical behavior, particularly their tensile and flexural strengths [40, 41, 45].

Silane is a chemical compound that can be represented by the formula SiH_4. As a coupling agent, silane is utilized to promote the attachment of kenaf fiber to a polymer matrix, which, in turn, results in an increase in the composite material's strength. It's believed that silane coupling agents lessen the amount of hydroxyl groups in cellulose at the fiber-matrix contact. The existence of water causes the hydrolysis of the hydrolyzable alkoxy group, which results in the production of

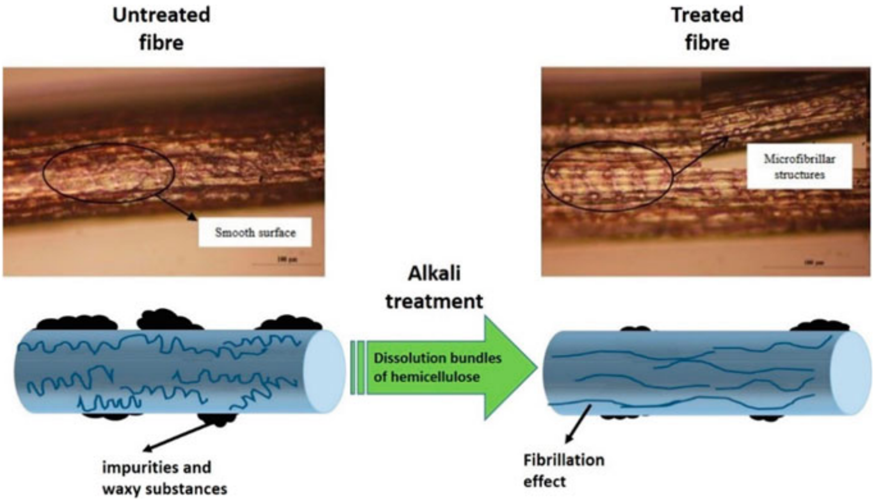

Fig. 3 Schematic diagram of alkali-treated natural fiber [44]

silanols. The hydroxyl group of the kenaf fiber then interacts with the silanol to produce covalent bonds, which are subsequently chemisorbed onto the surface of the fiber. The extension of the fibers is restricted by the silane-produced hydrocarbon chains (R), which, as a consequence of the transmission of hydrocarbon groups into the matrix, generate a tangled network [46]. The following reaction strategies are given in Eq. 5:

$$\text{Fiber} - \text{OH} + \text{R} - \text{SiOH}_2 - \text{R} \tag{5}$$

It was hypothesized that the hydrocarbon chains formed by the presentation of silane would influence the wettability of the kenaf fibers, which would then result in an increase in the chemical affinity of the polymer matrix. In addition, silane treatment improved the tensile strength of the composites, decreased the influence that moisture had on the properties of the composites, boosted adhesion, and as a result, raised the strength of the composites [40, 41, 45]. In recent years, researchers have begun to experiment with the silane treatment of kenaf fiber [39–42, 47]. Composites that had been treated with silane treatment exhibited exceptional interfacial adhesion among the kenaf filler and the polymer matrix.

The flexibility of kenaf fibers can be increased using a process called plant acetylation, which is a type of esterification. The cell wall has been stabilized through the widespread acetylation of wood cellulose, which has led to an increase in dimensional stability as well as environmental degradability. To acetylate lignocellulosic materials, the hydroxyl groups of the cell wall must be heated to extreme temperatures and then react with acetic or propionic anhydride (frequently in the absence of a catalyst) [46]. The reaction scheme is as described in Eq. 6:

$$\text{Fiber} - \text{OH} + (\text{H}_3\text{C} - \text{CO})2\text{O} \rightarrow \text{Fiber} - \text{O} - \text{CH}_3 + \text{CH}_3\text{COOH} \tag{6}$$

Before the kenaf fibers can be used, the acetic acid that is created as a byproduct needs to be removed first. The hydroxyl groups of lignin and hemicelluloses, both of which are amorphous materials, react with the reagent. On the other hand, the hydroxyl groups of cellulose, which are crystalline materials, hinder the diffusion of the reagent, which leads to extremely low reaction extents. It has been demonstrated that etherification results in an improvement in the dispersion of kenaf fibers within a polymer matrix, as well as an improvement in the dimensional stability and interface of the final composites [40, 41].

4.2 Bagasse Fiber-Reinforced Composite

Zheng et al. [48] investigated the effects of benzoic acid, which was used as an interface modifier, on the surface treatments of bagasse fiber (BF). PVC, benzoic acid, and other processing ingredients are dry-blended together in a blender before being subjected to compression molding. The results of the experiments indicated that the mechanical properties of the composite were significantly impacted by the ratio of PVC to BF, the concentration of benzoic acid, and the processing temperature. This was demonstrated by the orthogonal optimum technique, which was used to establish these relationships. The tensile strength of the composite was improved by the interface modification, however, the composite did not become more impact-resistant. As evidenced by SEM micrographs of impact-fractured BF/PVC composites and the finding that the addition of 10% benzoic acid increased the tensile strength of the BF/PVC composite from 38 to 52 MPa. The treatment of BF with benzoic acid significantly improved the dispersion of BF in the PVC matrix [48].

Vazquez et al. [49] described the handling and characteristics of bagasse fiber-polypropylene composites. Isocyanate, acrylic acid, mercerization, and cleaning with an alkaline mixture were the four separate chemical treatments that were used to the fibers in method to boost the interface linkage with the thermoplastic matrix. Infrared spectroscopy was utilized so that the effects that the various treatment procedures had on the chemical composition of the fibers could be examined. Photomicrographs taken with an optical microscope revealed that the procedure of mercerization marks in the establishment of a surface that is highly fibrillated. When untreated bagasse fibers are integrated into the polypropylene matrix composite, it was discovered that both the tensile strength and the elongation at the point of break are reduced. However, the tensile properties of the composite are enhanced through the processes of mercerization and isocyanate treatment. In addition to this, creep tests were performed on the composites that were being analyzed. The findings that were found to be the most successful were those that involved fibers that had been treated [49, 50].

Cao et al. [51] manufacture alkaline-treated biodegradable composites reinforced with bagasse fiber. The composites that are manufactured with alkali-treated fibers have superior mechanical qualities when compared to those that are made with composites that contain untreated fibers. The greatest amount of improvement was seen in composites that had fibers that had been treated with a solution containing 1% sodium hydroxide. The tensile, flexural, and impact strengths all improved by around 13%, 14%, and 30%, respectively. The treatment with alkali increased the aspect ratio as well as the strength of the fibers, which led to an advancement in the mechanical performance of the composites. Using a scanning electron microscope (SEM), researchers observed the rupture surface of composites. They found that surface alteration of the fiber had happened, which developed fiber-matrix adhesion [50, 51].

Martin et al. [52] utilized sugarcane bagasse that had been pre-treated with glycerol (40–80% (w/w)) at 190 °C, 10% biomass load, for 1 to 4 h. This process took place in the subsistence of NaOH or H_2SO_4 or without any chemicals. Following the application of the pre-treatments, the fibers produced exhibited increased levels of cellulose as well as varying degrees of xylan and lignin solubilization. When compared to pre-treatments that included H_2SO_4, those that included only the addition of NaOH and no other components resulted in a greater amount of cellulose being recovered than those that included any other components. In all of the treatments where H_2SO_4 was used, xylan solubilization was almost entirely accomplished; however, the results of the other studies varied depending on the concentration of glycerol used and the amount of time spent in the pre-treatment phase. The higher the concentration of glycerol, the greater the ease with which lignin might be dissolved. In every single one of the examined scenarios, the pretreatment resulted in an enhanced enzymatic hydrolysis of the cellulose. The greatest level of overall cellulose convertibility (85–94%) was achieved by doing pretreatments with glycerol alone, without using any other agents. Conversion decreased over time in acidic pre-treatments, but it marginally rose in the additional pre-treatments [50, 53].

A study by Monroy et al. [54] investigated sugarcane bagasse waste fibers reinforced poly (lactic acid) (PLA) matrix composite. The sugarcane bagasse fibers were first steeped in NaOH, then digested with NaOH, and finally blasted with steam before being impregnated with 1% maleated polyethylene. The use of scanning electron microscopy allowed for the discovery of surface morphological changes in the PLA matrix that were caused by fiber treatment/MAPE. Biocomposites containing MAPE were shown to have a greater rise in Young's modulus but a lower tensile strength. The results for biocomposites containing 10 and 20% bagasse have a flexural modulus of around 8000 MPa, which is significantly higher than the flexural modulus of PLA, which is just 3500 MPa. These improved qualities can be ascribed to an enhancement in the interfacial contact that exists among the modified sugarcane bagasse fibers and the PLA. The graph of X-ray diffraction reveals that the crystallinity of biocomposites that have been treated with MAPE and incorporated into a PLA matrix has a higher level of crystallinity than the crystallinity of the PLA matrix by itself [54].

4.3 Jute Fiber-Reinforced Composite

There are drawbacks associated with using jute fibers as reinforcements for composites because of the hydrophilic character of the fiber, its weak fiber-matrix interfacial adhesion, and its low wettability. Jute fiber must therefore undergo surface treatment in order to be usable. It is possible to improve the interfacial bonding and moisture resistance of the fiber matrix by removing impurities from the surface of the fiber, such as wax, hemicellulose, and lignin, and by adding additional chemicals. The chemical treatment of jute fibers has an effect on the chemical composition of the fibers as well as the surface qualities of the fibers [55, 56].

Sarker et al. [57] explored the qualities of elementary jute fiber as well as the impact of environmentally sustainable sodium bicarbonate treatment on the tensile properties, flexural properties, and interfacial properties of jute fiber/epoxy composites. They found that the elementary jute fiber had a high tensile strength and a low flexural strength. When compared to fibers that had not been treated, tensile strength was enhanced by 56% and modulus was enhanced by 50% when 10% sodium bicarbonate was applied. Jute fibers that were treated with 10% sodium bicarbonate and an epoxy matrix exhibited a 175% improvement in their interfacial shear strength (IFSS). When compared to untreated jute fibers, composites containing 10% sodium carbonate-treated jute fibers showed an increase in flexural strength of over 32%. This notable expansion of alkaline-treated jute fibers is due to the elimination of hemicellulose from the interfibrillar area of jute fiber, which boosts the load-bearing capacity of individual fibers and further raises the fiber content in composites [57]. The alkaline treatment removes hemicellulose from the interfibrillar area of jute fiber, which results in this great improvement.

IFSS is recognized as one of the defining parameters for composites' bending, damage tolerance, and compressive qualities. The value of the IFSS can be determined by applying the load–deflection curve from the microbond test to the debonding force in order to convert it to shear stress. A low IFSS value is the result of the presence of unwelcome impurities on the surface of the untreated jute fibers, which causes the chemical interaction between the jute fiber surface area and the epoxy resin to be poor and results in a poor IFSS value. During microbond testing, it is predicted that a fracture will take place in the composites' weak cohesive zone (cementitious layer and epoxy). The presence of this thin layer of cohesive cement precludes the fiber from sustaining any further loads throughout the test; as a result, the fiber's outermost layer and the epoxy resin readily separate [57].

The sodium bicarbonate solution is used to remove the polysaccharides and other surface defects from the jute strands. The normal alkaline treatment can be replaced with a bicarbonate treatment, which can dissolve the hemicellulose that is responsible for the stress concentration of jute fibers when they are loaded. In spite of the fact that bicarbonate is not designed to remove lignins, a negligible amount of lignin is removed as a result of this treatment as a result of the high concentration of sodium bicarbonate (10%) that is utilized. After the removal of the polysaccharides that were stated earlier, the diameter of the jute fibers shrunk significantly and became more consistent. In addition to this, the surface of the fiber is smooth, and it has an appearance that is less ridged. In addition, the larger diameter of the untreated fiber suggests the presence of a greater number of cellulosic components. The fibres have a propensity to move closer together after the bicarbonate treatment. This is due to the fact that the bicarbonate treatment significantly reduces hemicelluloses while also causing the chain of cellulose fibrils to form new hydrogen bonds. Because of this, fibrils are able to reorganize themselves in a denser manner, which leads to a denser packing of fibers and makes it possible for a greater amount of stress to be generated during tensile loading [57].

The fine structure of cellulose and other polysaccharides in hot-alkali-treated jute fibres is schematically depicted in Fig. 4a–c at various alkali concentrations. Figure 4a–f, on the other hand, show SEM images of untreated, alkali-treated jute fibers [58]. Untreated jute fibres have a pectin, wax, and impurity-coated surface morphology, which may limit the area of interaction between the jute fibres and the resin. The pectin, wax, and impurities are eliminated by the use of an alkaline treatment, which also results in the formation of a variety of folds, gaps, and minute spaces. Jute fiber receives an alkaline treatment, which results in the fiber becoming clean and coarse. The treatment of cellulose with alkali improves its crystalline structures, shortens the space between adjacent cellulose chains, produces hydrogen bonds between adjacent cellulose chains, and makes the fiber more robust [59].

4.4 Coir Fiber-Reinforced Composite

Coir fibers have exceptional mechanical qualities, such as the capacity to enhance the tensile intensity of polymer resin and the ability to achieve the highest level of elongation among all natural fibers that are currently known. Contrarily, polymer resin is hydrophobic, whereas coir fibres are hydrophilic. Coir fibres have hydroxyl groups on their surface that can bind with water molecules to form hydrogen bonds with them. These hydrogen bonds limit mutual penetration and lead to weak interfacial bonding capacity, both of which have a negative impact on the mechanical properties of composites [60, 61]. The compatibility of coir fibres with polymer resin matrix is currently being improved through the conduct of numerous studies.

Shrivastava et al. [62] examined how coir-fiber reinforced epoxy resin composites' tensile strength was affected by alkali treatment. The results showed that after the alkali treatment, the coir fibres were more securely attached to the matrix, and the tensile strength of the composites was significantly higher than that of earlier composites [62]. In order to increase fiber-matrix adhesion and, as a result, strengthen the interfacial bonding between coir fibres and the epoxy resin matrix, anhydride surface modification involves reducing the number of hydroxyl groups on the fibre surface and swapping them out for anhydride group [63, 64]. This is done in an attempt to promote fiber-matrix adhesion.

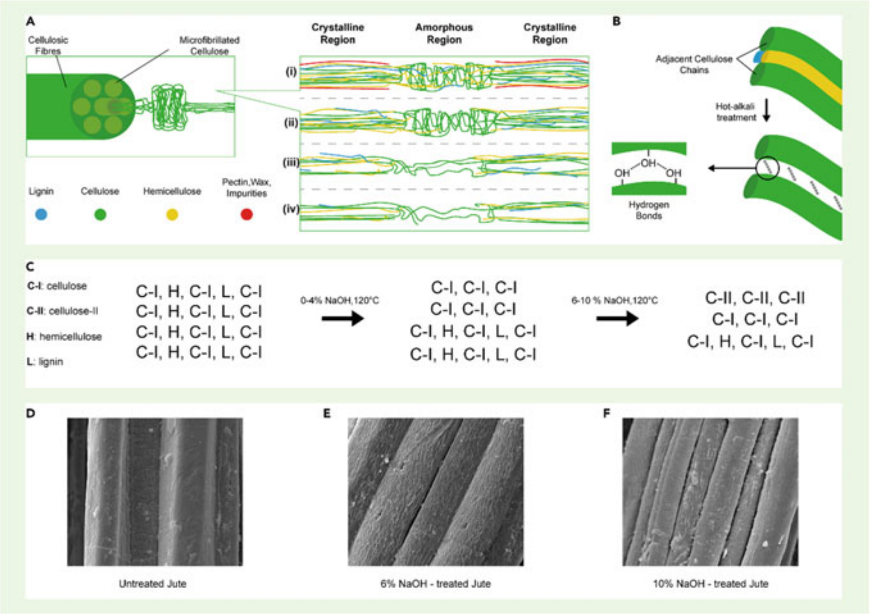

Fig. 4 The alkali treatment of jute fiber **a** A diagram showing the fine structure of cellulose and other polysaccharides in hot-alkali-treated jute fibers (i) untreated, (ii) 2% NaOH-treated, (iii) 4% NaOH-treated, (iv) 6%–10% NaOH-treated, **b** neighboring cellulose chains, and **c** variations in the amounts of cellulose, hemicellulose, and lignin. Surface SEM images of jute fibers: **d** untreated, **e** treated with 6% NaOH, and **f** treated with 10% NaOH [58]

Ru et al. [65] looked into the interfacial adhesion that exists between coir fibers and an epoxy resin matrix. The modification of coir fibers was accomplished through the use of microwave treatment, alkaline treatment, acetic anhydride modification, 3-aminopropyltriethoxysilane modification, and their respective practical combination treatment methods. Experiments testing the pullout of a single fiber were carried out on the coir fiber pullout specimens described previously. The effect of a variety of treatments on the characteristics of coir fibers was investigated using scanning electron microscopy (SEM), Fourier transforms infrared (FTIR), and X-ray diffraction (XRD). The results of the proposed method of estimation suggested that the combination of alkali treatment and surface modification with 3-aminopropyltriethoxysilane could enhance the interfacial bonding ability between coir fibres and epoxy resin. Pullout energy was calculated as 40,300 Nmm and the interfacial shear strength as 6,728 MPa. The primary investigation demonstrates that the approach can establish both mechanical interlocking and chemical connections at the contact in order to promote interfacial adhesion [65]. Figure 5 shows the mechanism of the interface between the coir fibre and the matrix following the treatment.

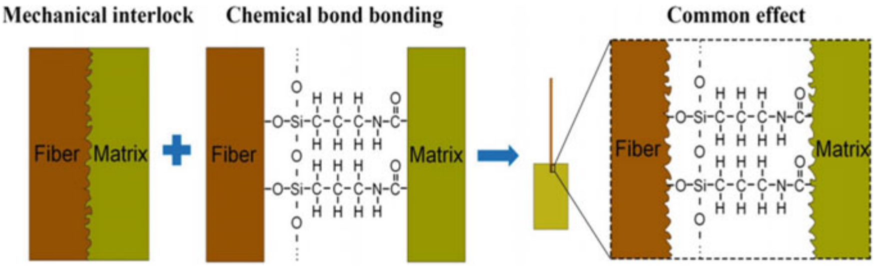

Fig. 5 Alkali treatment and modification of 3-aminopropyltriethoxysilane together improve the mechanism of interfacial characteristics [65]

4.5 *Sisal Fiber-Reinforced Composite*

Ayalew et al. [66] examine the manufacture of sisal-reinforced polyester composites. The effects of the concentration of NaOH (2, 6, and 10%), the length of time that the fibers were immersed in the solution (24, 48, and 72 h), and the ratio of sisal to polyester (weight per weight) were studied. The findings indicate that the mechanical characteristics of sisal plant fibers treated by various concentrations of NaOH are significantly improved in comparison to those of sisal fibers that have not been treated in any way. According to the findings of the FT-IR analysis, the elimination of lignin and hemicelluloses as functional groups suggested either the lack of hemicelluloses or a progressive decline in peak intensity as the concentration of alkali increased. After 48 h of treatment with a 6% NaOH solution, the composite material's tensile and bending strengths were brought up to their maximum levels. Alkalization increased interfacial adhesion and boosted mechanical characteristics within the parameters of the conditions that were allowed. Following the extraction and treatment of fibers, composites were generated by interlocking fibers in matrices at concentration ratios of 20: 80, 30: 70, and 40: 60 fiber-to-matrix weight ratios respectively. When using a fiber-to-matrix ratio of 30:70, the best tensile and bending strengths were found to be 44.003 MPa and 50.81 MPa, respectively. The lower water absorption demonstrated by treated fiber composites can be attributed to the increased surface area of fiber contact with the matrix, which is less permeable to water than natural fibers. At 72 h, a 6% solution of NaOH and a 20:80 fiber-to-polyester ratio of 3.194% decreased water absorption. Alkaline treatment of sisal fiber modifies the fiber surfaces considerably and is examined for all mechanical parameters [66].

Kaewkuk et al. [67] conducted comparative research on the influence of fiber treatment (alkalization and heat treatment) and the inclusion of a compatibilizer (maleic anhydride-grafted polypropylene, MAPP) on the properties of PP composites. In their study, they found that the properties of PP composites were affected by both factors. The water resistance, cellulose decomposition temperature, and mechanical properties of the PP composites were all significantly improved as a result of the fiber treatment and the addition of MAPP. SEM micrographs show that the interfacial modification increased the adhesion between the fiber and the PP matrix. This was demonstrated by the interfacial modification. The mechanical characteristics of sisal fiber/PP composites were improved by alkalization and heat treatment, respectively. The most significant advancement in the PP composites' improved mechanical characteristics came about as a result of the incorporation of MAPP. The tensile strength and modulus of PP composites increased with increasing fiber amount. On the other hand, their impact strength and elongation at break decreased as the fiber content grew. In addition, the amount of fiber present in PP composites led to an increase in their capacity to absorb water [67].

The scanning electron micrographs of composites incorporating sisal fiber and PP are displayed in Fig. 6. Figure 6a depicts the surface of the fracture that occurs on an untreated sisal fiber/PP composite that has a substantial gap between the fiber and the PP. The interfacial changes, which are shown in Fig. 6b–d, increased the interfacial adhesion between the fiber and the PP matrix. This occurred because the gaps between the fiber surface and the PP matrix shrank. The mechanical characteristics of the PP composites were improved as a result of the interfacial modifications. The mechanical characteristics of PP composites treated with either alkalization or heat are the same. This indicated that the various techniques for treating fibers were comparable. MAPP proved to be the component with the greatest potential

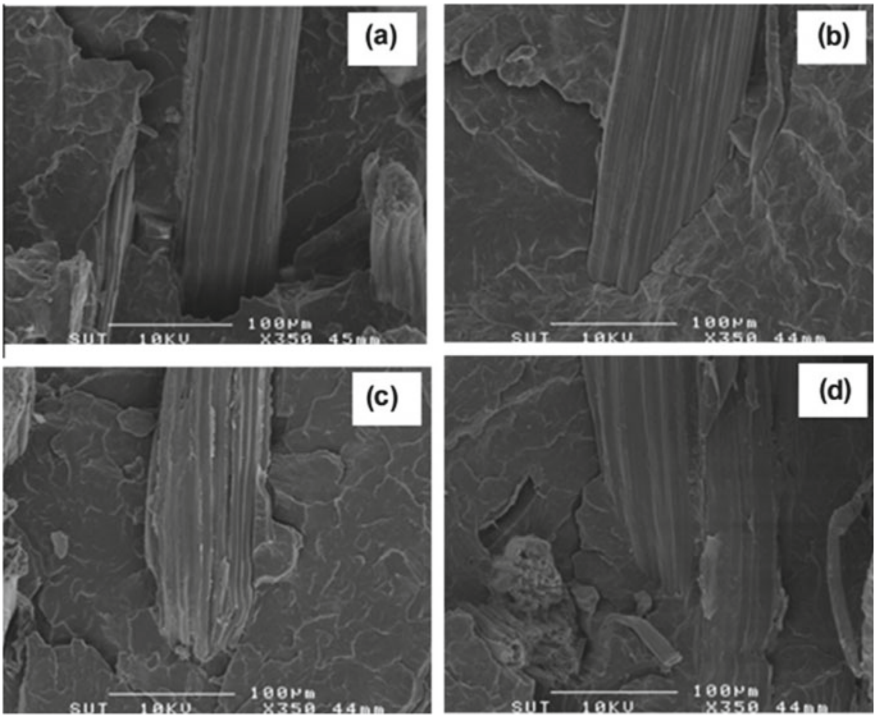

Fig. 6 SEM micrographs of the surface of UT/PP (**a**), AT/PP (**b**), HT/PP (**c**), and UT/PP/MAPP (**d**) composite at 20 wt.% fiber content [67]

for improving the mechanical properties of PP composites. When the fiber content of the PP composites was raised, the tensile strength and Young's modulus of the composites increased, but their elongation at rupture and impact strength declined. According to micrographs taken using a scanning electron microscope (SEM), the interfacial changes improved adhesion between the fiber and the PP matrix [67].

5 Summary

This chapter has examined the significance of surface treatment processes in a variation of polymer matrix composites reinforced with natural fibers. The text discussed the various surface treatment effects on the enhancement of composite material qualities. Due to the wide diversity of natural fiber types, which influence its chemical composition, the treatment conditions vary depending on the reinforced fiber chosen. As a result of this brief analysis, the subsequent concerns might be emphasized for a deeper understanding of the influence of surface treatment conditions on the improvement of the mechanical properties of diverse natural fiber-reinforced polymer composites.

1. Mercerization or alkali treatment is one of the most frequent procedures employed to modify the surface of fibers. Moreover, compared to other chemical treatment approaches, this treatment was regarded as the easiest and most cost-effective option.
2. In this brief review, it was made abundantly evident that, despite the vast amount of published research on natural fiber mercerization treatment, there have been very few studies on interaction and optimizing the mercerization treatment parameters.
3. The most common surface modification was alkaline treatment. Despite the use of the same types of reinforced fiber, different surface treatment processes may result in varied mechanical property values for the finished composite.
4. Therefore, there is a great need for additional research focused on the primary impact and interaction effect of others surface modifications to improve the mechanical properties of natural fiber-reinforced composites.

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Effect of Fiber Dosage and Chemical Treatment on the Vibrational Behavior of Luffa/ USP Composite

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Abstract In this work, the vibrational behavior of luffa cylindrica/USP composites has been addressed. The effect of fiber dosage with difference fiber content from 30, 40, 50% and chemical treatment (NaOH) are analyzed. The specimens are fabricated with compression molding technique with pressure of 17 MPa. For the production of composite specimens, untreated and NaOH treated fiber were used as reinforcement and unsaturated polyester resin (USP) used as matrix. The prepared specimens are cut as per the ASTM standard, and mechanical and vibrational tests were conducted. The experiential nodal analysis is used to fine the natural frequency and damping of the composites. The results depicts that the increase in fiber dosage started to improve the mechanical properties of the composite beam. The NaOH treatment showed the improvement in mechanical and damping of the composites. The 50% fiber dosage showed the good tensile and flexural properties of the composites. The interfacial mechanism of the composites was interpreted in the SEM.

Keywords Luffa · Natural fiber · USP · Natural frequency · Damping · Mechanical properties

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1 Introduction

Now a days the natural fiber have been much more attention by the researchers because of its good performance towards industrial applications. The natural fiber reinforced composite (NFRC) provides high specific strength, low cost, easy availability, and bio degradability [1–3]. The most commonly planet fiber used in various low load applications like mats, fishnets, mats and wall coverings are banana, jute, sisal, hemp, palmira, ramie, coir, flax [4]. Kumar et al. [5] examined that the impact of fiber dosage and sequence of sisal and banana fiber composites on mechanical and vibration characteristics and reported that maximum value of mechanical properties found on the 40% fiber dosage. In addition to that, the fiber dosage may affect the natural frequency of the composites. Senthilkumar et al. [6] analyzed that impact of length of fiber and fiber dosage on mechanical behavior and vibrations properties on banana/sisal composites and also they observed that increase in fiber dosage stared to enhance the mechanical and damping ability. Rajini et al. [7] researched on chemical modification effects on dynamics mechanical analysis along with vibration behaviors on coconut sheath and nano clay/USP hybrid composites and concluded that the vibration properties have been improved in 3 wt.% addition of clay in hybrid composites. Senthilkumar et al. [8] investigated stacking sequence impact on the vibration properties of short banana and woven coconut sheath/USP composites with effect of chemical treatment. Kalusuraman et al. [9] earlier concluded that the surface treatment impact on friction co-efficient of luffa/USP composites. Kalusuraman et al. [10] studied about the DMA studied on luffa/USP composites and reported the fiber loading enhances the storage modulus. From the literature, It can be revealed that the most of the researchers have done the vibration behaviors of the many natural fiber composite. But the natural behaviors of luffa/USP composite are scanty. On this context, the present work describes the vibration and mechanical properties of luffa/USP composite by varying fiber dosage with effect of surface treatment.

2 Experimental Details

In this study, luffa fibers are used supplied from local market. Unsaturated polestar resin, accelerator, and catalyst are supplied by Vasivibala resins Pvt. Ltd., Chennai, Tamil Nadu, India.

2.1 *NaOH Treatment*

The fibers were permitted to submerge in the alkali solution which was made by addition of 40 g of NaOH in 1000 ml distilled water for an hour. After that the fibers were cleaned thoroughly by using the distilled water, then it would be in the hot oven for 60° for an eight hours.

2.2 Fabrication of Composite Material

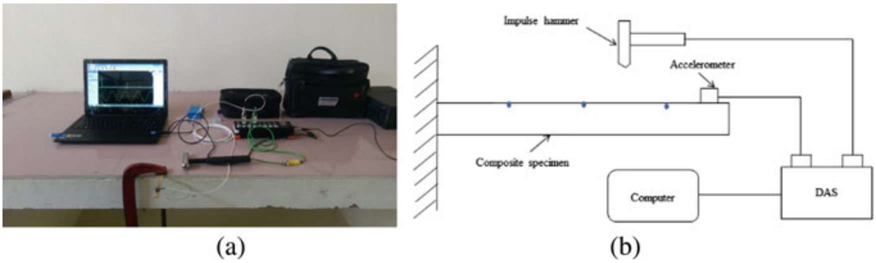
Luffa fiber with treated and untreated fiber composites was fabricated by using a metal die of 300 × 125 × 3 mm. The fibers were placed in the stacking order on the mold and it was pre-compressed by using a compression moulding machine. Catalyst and accelerator each 1.5% was used to speed up the chemical reaction. The catalysed resin was poured on the fiber kept in mold. The closed mould was compressed by machine with an optimum pressure of 17 Mpa. The wax was used in the mold as a releasing agent. Catalyst and accelerator each 1.5% was used to speed up the chemical reaction. The entire set up was allowed to curing in the normal room temperature for 24 h. Then the samples were cut as per the ASTM.

2.3 Mechanical Properties

The Instron Testing machine (series 3382) was used to conduct the tensile test to find the stringth of specimen as per the ASTM D3039. The specimen with 120 × 15 × 3 mm dimension were cut from the fabricated plate. The specimens were tested with 600 mm gauge length and also the cross head speed of 3 mm/min. The flexural test was carried as per the ASTM: D790-10. The Charpy impact test was perfomred as per ASTM: D256-10 to find the impact strength. The average of 5 specimens was used in each test and reported.

2.4 Modal Analysis

The Modal analysis is a very vital method to examine the natural characteristic such as vibration and damping factor of the material. It is very significant to study the vibration and noise control of the materials which is being used in the automobile sector, and even the sports sector. There two technique available to study modal analysis of the material such as impact hammer method and vibration shaker method. None of the difference was expected from these two methods [15]. The impact hammer technique is appropriate for the light weighted material. So the impact method was used to find the natural frequency of the material, at the end of the plate of the specimen an accelerometer was attached (Kistler model: 8778A500) using wax. Sharp hardened tip of impact hammer (Kistler model: 9722A500) was used to hit the specimen (120 × 15 × 3 mm) at three equally space provided in the speciemen. The displacement signal has been noted in PC using the accelerometer with data acquisition



(a) (b)

Fig. 1 **a** Experimental set up of free vibration analysis, **b** line diagram

system (DEWE 43, Dewetron Corp., Austria) and ICP conditioner (MSIBRACC). The two separate connectors for taking the picture have been utilized to find the output signal and response signal from the accelerometer and hammer respectively for the individual specimen. Figure 1 shows the experimental set up for vibration test.

2.5 *Damping Factor*

Damping is the energy dispersion properties of a material when it subjected to under cyclic stress. Damping factor is used to fine the Dynamic characteristics of composite specimen. The following equation is used to get the values of damping of the specimen by frequency response function (FRF) curve. It could be obtained from the Fast Fourier Transform (FFT) analyzer.

$$X_n/X_{n+1} = e^{\varepsilon * \omega n * Td} \quad (1)$$

where, X_n—amplitude of 1st peak, X_{n+1}—amplitude of 2nd peak, ε—Damping factor, ω_n—natural frequency, Td—time interval. Figure 2 shows the general line diagram of damping.

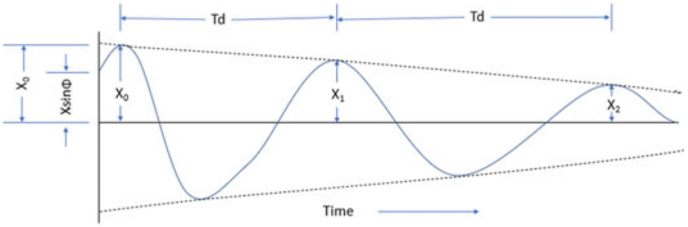


Fig. 2 Line diagram for damping

3 Result and Discussion

3.1 Mechanical Properties

Table 1 depicts the combined effect of the fiber dosage and surface enhancement of luffa fiber/USP composites. An increasing tensile strength trend is observed in the UT, NT composites. The NT 50 composite holds higher tensile strength among all the composites. The lower values found for UT 30 composite was 22.951 MPa. An increasing flexural strength was found as a result of an enhancement in fiber dosage. The higher flexural strength was found in 50% fiber loading for UT and NT composites. The maximum value of 45 MPa was found for the flexural strength of NT 30 composite. The lowest value of 25 Mpa was found for the flexural strength of UT 30 composite.

The strong adhesion between the matrix and fiber is noticed for NT composites as in Fig. 3a. The thick interface can be noted which attributes the increase in the tensile strength of specimen. Figure 3b depicts more fiber pull out, a gap in interfacial and more delamination which associates the lower tensile strength of the composite.

Regarding the impact strength, they tend to increase as a rise in fiber loading. But in UT composites, a little variation was found due to unavoidable porosity in the composite. The porous nature of the individual fiber promotes the impact strength of the composite. However, other factors like interfacial adhesion and matrix-fiber bonding will improve the impact strength of the composite. So, the chemically treated composites produced the good results than the UT composites. Form Fig. 4, more fiber pull leads to an increase in energy dissipation, resulting in high impact strength of 29.32 kJ/m^2. More separated fiber can be noted during the metallographic inspection. Poor wetting and less interfacial bond between the fiber and matrix for UT30 composite depicts a less impact strength of 5.83 kJ/m^2.

Table 1 Mechanical properties of luffa/USP composites

Composites	UT30 (untreated 30% fiber dosage)	UT40 (untreated 40% fiber dosage)	UT50 (untreated 50% fiber dosage)	NT30 (NaOH treated 30% fiber dosage)	NT40 (NaOH treated 40% fiber dosage)	NT50 (NaOH treated 50% fiber dosage)
Tensile strength (MPa)	22.951	25.344	30.022	39.765	41.190	46.479
Flexural strength (MPa)	25.01	29.99	31.59	45.06	43.06	45.02
Impact strength (kJ/m^2)	5.8317	15.4667	12.5468	5.92534	11.6965	29.32468

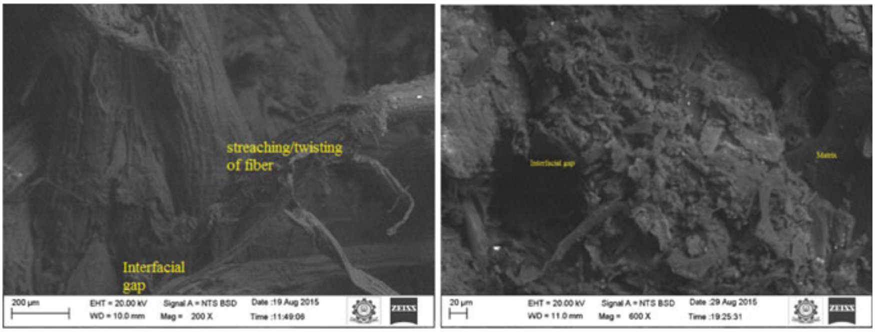
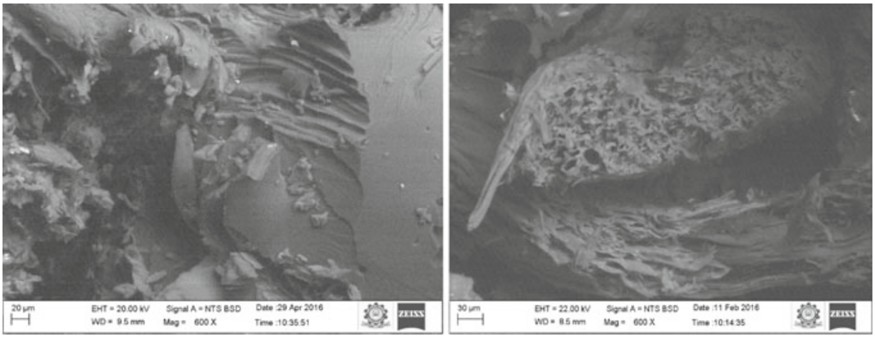


Fig. 3 Fractured surface of tensile specimen **a** NT 50, **b** UT 30



(a) (b)

Fig. 4 Fractured surface of impact specimen **a** UT 30 **b** NT 50

3.2 Natural Frequency

The NFRCs depends on varies factor such as fiber dosage, fiber length, fiber& matrix bonding, so it is hard to calculate the NF of the composite. In this work, three modes of the composite were discussed and reported for UT and NT composites. A contact force was applied at one corner of the cantilever structure of the composites with the aid of piezoelectric impact hammer. A constant force was given at five various spot on the composite which induced the static deformation as it was validated by the same force peak values via the oscillation rate of the force changed. The triggered signal was obtained with the help of the accelerator, connected at the corner of the place. The frequency response function is the common method for measurement of such analysis ie. output response to input excitation force. The Fast Fourier transform (FFT) analyser is used to acquire this measurement. Using a FFT algorithm, the measured time data can be transferred time domain to frequency response function in signal processing analyzed and system software. The FRF function peaks are produced at the resonance frequency of the system.

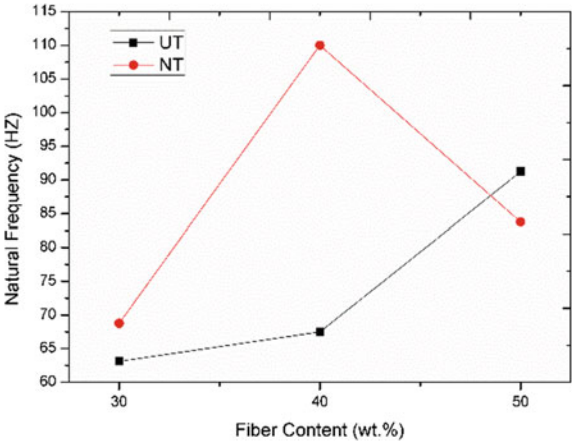


Fig. 5 Effect of dosage on natural frequency of composites (mode-1) for UT and NT luffa composites

Table 2 Natural frequency of the Luffa/USP composite

Natural frequency (Hz)	Composite types					
	UT 30	UT40	UT 50	NT30	NT40	NT50
Mode1	63.13	67.5	91.25	68.75	110	83.75
Mode2	418.1	477.5	560.63	470	681.3	530
Mode3	1180.6	1352.5	1495.6	1014.4	1804.1	1458.1

There are three modes of deformation available in the structure/composites such as bending (mode-1), twisting (mode2) and second bending (mode-3). The first three modes of UT 30 are 63.23 Hz, 418.1 Hz and 1180.6 Hz respectively. Figure 5 shows that the first mode of the natural frequency of luffa UT and NT composites.

The UT composites shows the increase in natural frequency trend and increase in the fiber dosage. The higher natural frequency is noted for both UT and CT composites at 50% fiber loading. A notable difference and huge difference are noted for 30%, 40% and 50% respectively. An increasing trend of natural frequency is noted for the UT composites. But in the case of NT, 50% fiber loading leads to a decrease in the natural frequency. It might be due the agglomeration of the fiber which causes the weak fiber matrix bonding. Nevertheless, increase in the fiber loading increases the stiffness of the composite which improves the natural frequency of UT composites. Table 2 shows that the natural frequency of UT and NT composites.

3.3 Damping Factor of Composites

Figure 6 shows the frequency response of the NT 50 composite. It depicts the first mode treated and untreated luffa/polyester composites. Generally, the higher fiber

Fig. 6 Effect of fiber dosage, and surface treatment on damping behavior

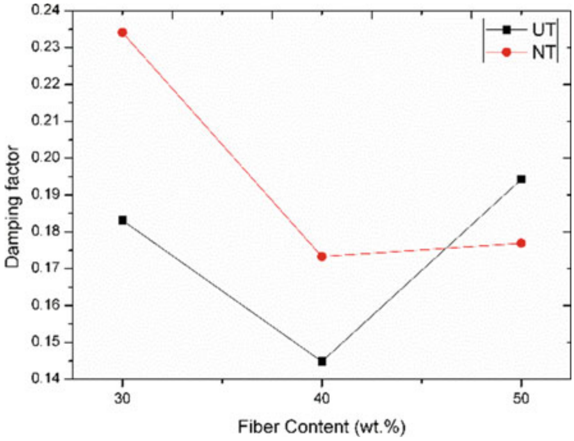


Table 3 Damping of the Luffa/USP composites

Damping	Composite types					
	UT 30	UT40	UT 50	NT30	NT40	NT50
Mode1	0.183134	0.144839	0.19427	0.234093	0.173269	0.176912
Mode2	0.026788	0.023385	0.030698	0.034874	0.028158	0.026168
Mode3	0.012412	0.008812	0.011158	0.012647	0.009631	0.009460

loading will lead the higher damping ratio since their viscous elastic behaviour in nature. However, it is observed that higher fiber dosage gives high damping. It is in contradiction for luffa/polyester composites. The porous structure of the fiber may also be the reason for higher damping. The measured damping values of the UT and NT composites are very similar in mode-1 rather than mode-2 and mode-3. From the Table 3, it is clear that other than the fiber dosage, interface stiffness is also the most significant factor on the damping behaviour. The NT composite holds the higher damping than UT composite. The surface treatment improves the surface roughness which increases the interface bonding.

4 Conclusion

This work reports the effect of fiber dosage and chemical treatment on the vibrational behavior of Luffa/ USP composite. The following conclusions were drawn:

- The luffa /USP composite were successfully produced by using the compression moulding process by varying fiber dosage.
- Better mechanical properties were found for the NT composite in comparison with the UT composite.

- The higher fiber loading (50%) gives significant improvement on tensile and flexural properties of the composites.
- The optimum fiber dosage was observed as 50% for luffa/polyester composites.
- The maximum natural frequency was found for NT40 composite. In addition, the NT40 composite offers higher damping values among all the types of composites.

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