REVIEW



A Comprehensive Review on Epoxy Biocomposites Based on Natural Fibers and Bio-fillers: Challenges, Recent Developments and Applications

Fazal Maula Khan¹ · Ahmer Hussain Shah² · Shuo Wang¹ · Shah Mehmood¹ · Jun Wang¹ · Wenbin Liu¹ · Xiaodong Xu¹

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Abstract

Natural fiber-reinforced polymers remained a hot topic of interest in material sciences over the last two decades. Such fibers are appealing in composite materials due to their renewability, low density, better specific strength, biodegradability, accessibility, and low cost. Polymers reinforced with natural fibers provide improved mechanical performance at a lower cost and could be the best alternative to synthetic fibers. Bio-fillers have gained much attention nowadays due to their cost-effectivity and the ability to modify base materials in the composite structure. Natural materials reinforced epoxy composites have a high capacity due to their eco-friendliness, economic feasibility, and technical viability. However, some parameters directly influence desired product performance. This review discusses various properties of natural fibers and their impact on the overall performance of natural fiber-based epoxy composites. It summarizes the recent research on natural fibers/bio-fillers reinforced epoxy biocomposites.

Keywords Biocomposites · Natural fibers · Bio-fillers · Reinforced epoxy composites

Introduction

Due to various environmental benefits, researchers are working to produce renewable composites that are fully or partially bio-based [1]. A bio-composite is a composed of a polymer matrix with natural fibers and reinforced bioparticles. Synthetic fiber-reinforced polymer composites have been widely used recently for various technical applications due to their high strength-to-weight ratio. Nevertheless, they have drawbacks like the high cost of initial material, increased energy demand, non-biodegradability, and environmental impact. Natural fibers are considered to be the best substitute for synthetic fibers. Natural fiber-reinforced composites are less expensive and more environmental friendly than synthetic fiber composites [2, 3]. Natural fiber-based composites consist of natural fiber in the polymer matrix. By 2024, the market for natural fiber-reinforced composites is predicted to reach \$10.89 billion. The natural fiber-reinforced polymers have found successful applications in automobiles, aircraft, constructions, electronics, and other industries to replace the internal and external work because of their reduced price, high stiffness, strength, degradability and reduced weight [4–7]. Load-bearing components for structural and infrastructural purposes have been made from natural fiber composites. A few examples of such fibers include multipurpose panels, beams, roofing, storage tanks, and pedestrian overpasses [8].

Two main types of polymers include thermoplastics and thermosets. Polyester, phenolic, and epoxy are the most commonly used thermosetting resins [9, 10]. Epoxy-based composite materials possess good mechanical properties, super adhesiveness, high specific strength, good heat, and solvent resistance at a lower cost and are widely used in load-bearing applications. Currently, there is a great need to understand the properties of natural materials to fulfill

Xiaodong Xu xuxiaodong@hrbeu.edu.cn

¹ Key Laboratory of Superlight Material and Surface Technology, Ministry of Education, College of Materials Science and Chemical Engineering, Harbin Engineering University, Room 534, Jichu Building, 145# Nantong Street, Nangang District, Harbin 150001, China

² Department of Textile Engineering, Balochistan University of Information Technology, Engineering and Management Sciences, Quetta 87300, Pakistan

engineering requirements. The use of natural fibers-based epoxy composites for structural materials has received great attention in the last two decades [11, 12]. Researchers are therefore continually working for more practicable and durable development and industrial application of biocomposites. This review encompasses various parameters affecting the performance of natural fibers and specifically discusses the recent research done on bio-fillers and natural fibers-based epoxy and hybrid composites.

Challenges Using Natural Fibers for Composites

Effect of Chemical Composition

Natural fibers consist of proteins, lignin, waxes, cellulose, pectin, and hemicellulose. Table 1 summarizes the chemical composition of different natural fibers [13–15]. The chemical composition of plant fiber, and resultant the composites has a significant impact on their properties. Cellulose and hemicellulose have a hydrophilic tendency, indicating that plant fiber has high moisture content and, as a result, the composite has low moisture resistance [16]. The primary parameters to achieve excellent performance are a high degree of polymerization, a higher cellulose content, a smaller microfibrillar angle, and a higher cellulose crystallinity. Fibers' Young's modulus and tensile strength typically increase as cellulose content rises. The stiffness of fibers is determined by the microfibrillar angle. If the microfibrils angle is low, fibers are more ductile, whereas fibers with high microfibrils angle will be inflexible and stiff with high tensile strength. Hence, all these factors are taken into account while selecting plant fibers as reinforcement in structural applications. Bast fibers have a low microfibril angle, a high crystallinity, and a high cellulose content as they support the plant's stalk [17].

Interaction between Fiber and Matrix, Adhesion Problems

The majority of natural fibers contain a cellulose-rich core as well as waxes, fats, lignin, pectin, and hemicellulose. Lignin, oils, and wax cover the outer surface that serves as a cementing layer and prevents the formation of powerful interphase. It renders the surface of fiber highly hydrophilic and incompatible with hydrophobic polymer matrices. Therefore, the main objective is to improve the interphase by removing this cementing layer. Removal of this layer improves mechanical interlocking by increasing interfacial shear strength along with fiber wettability [18]. The fiber/matrix interface and fiber surface wetting property are critical in determining the mechanical properties of composites. The flexural strength, tensile strength, and toughness of composites are all affected by fiber wettability [19–22]. There are various physical and chemical techniques to increase the interfacial strength of a composite.

Mercerization of natural fibers is commonly used to increase the compatibility of natural fiber and polymer resin. The alkaline treatment exposes the amount of cellulose on the fiber surface, enhances surface roughness, and increases the number of possible reaction sites, hence providing a better mechanical interlocking [23]. The higher the surface energy of plant fibers, the greater the fiber wettability, and higher the surface roughness, the better mechanical interlocking and the greater the fiber epoxy matrix adhesion. Furthermore, the combination of alkali, organosilane, and epoxy dispersions also improves the jute fiber-epoxy adhesion strength. The interfacial shear strength of jute/epoxy composites increases significantly by up to 40% [18]. Acetylation increases the fiber's surface roughness, resulting in enhanced mechanical interlock with the matrix [24]. The maleated coupling treatment modifies the polymer matrix and fiber surface, augmenting interfacial bonding between matrix and fiber [25, 26]. Silanes increase the hydrophobicity of natural

Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectins (%)	Waxes (%)
Jute	61–71.5	13.6–20.4	12–13	0.2	0.5
Flax	70–73	18.6-20.6	2.2	2.3	1.7
Hemp	70–74	17.9–22.4	3.7-5.7	0.9	0.8
Kenaf	45-57	8–13	8-13	3–5	0.8
Sisal	66–78	10–14	10–14		2
Banana	63–64	10	5		_
Abaca	56-63	20–25	12-13	1	
Cotton	95	2	1		0.4
Coir	32–43	10-20	40-46	3–4	4
Bamboo	26–43	30	28		_
Bagasse	55.2	16.8	25.3		-

Table 1Chemical compositionof various natural fibers [13–15]

fibers, thus improving the strength of natural fiber reinforced composites [27]. UV treatment induces the polarity of fibers and improves the bending strength of epoxy matrix composites up to 30% [28]. Plasma treatment enhances the surface roughness of fiber, consequently increasing interfacial adhesion [21]. Plasma treatment also improves bending strength and interlaminar shear strength in natural fiber composite up to 30% and 35%, respectively [29]. Chemical treatments remove the waxy substances such as lignin and hemicelluloses from the fiber surface, leaving the fiber surface rough (Fig. 1). This roughness boosts mechanical interlocking and promotes fiber-matrix adhesion, resulting in better mechanical performance [30].

Fiber Strength Variation

One of the major problems with natural fibers is that their qualities vary even within the same type of fiber. The properties of natural fibers change significantly depending on chemical composition and structure, harvesting period, growing conditions, extraction techniques, treatments, storage methods, and even testing methods/standards. As previously stated that due to its inherent nature, chemical composition strongly affects fiber properties. In addition to cellulose content and microfibrillar angle, other structural parameters such as cell dimensions, and defects also play an important role in the determination of fiber properties [31]. The harvest season has an impact on fiber properties as well. Harvesting in the autumn and winter is best for mechanical properties [32]. The studies have revealed that the tensile strength of hemp fiber varies during the flowering stage and increases up to 114 days of growth [26]. The retting technique also influences the chemical composition,

structure, and performance of the natural fiber. Biological retting produces high-class fibers, but it increases environmental pollution as this method generates a large amount of wastewater, making it unsuitable for most commercial applications [15]. The tensile strength of elementary flax fiber varies depending on the isolation process and ranges from 1500 to 1800 MPa. The strength of flax fiber extracted by hand was found 20% higher than that of flax fiber extracted by machine [33]. Other factors influencing the natural fiber properties include fiber diameter and cross-section area. The difference in determining the cross-sectional area of plant fibers causes a significant variation in the strength and modulus of the same plant fiber. The tensile modulus of a single natural fiber decreases as the diameter of the fiber increases [14].

Moisture Absorption

Due to abundant hydroxyl groups, the moisture resistance of natural fibers is poor. They absorb moisture from the environment until equilibrium is achieved. This moisture absorption in hydrophilic natural fibers has a significant impact on their physical and chemical properties, resulting in dimensional variations, changes in mechanical and chemical performance, and composite failure. The amount of moisture absorbed by the fibers influences their volume in the composite to reinforce natural fiber in a polymer matrix. Secondly, polymerization at temperatures above 100 °C vaporizes the water contained within the natural fiber. These effects cause internal stresses in the composite which can lead to a significant decline in the preliminary properties of the composite. Assarar et al. [34] fabricated epoxy composites out of glass and flax fiber and compared the effects of



Fig. 1 Scanning electron microscopic images of **a** untreated jute fiber, **b** alkali treated jute fiber, **c** silane treated jute fiber, and **d** alkali and silane treated jute fiber [30] water aging on both composites. They claimed that moisture absorption of flax fiber composite was excessively high when compared to glass fiber reinforced composite. The saturated weight gain of flax fiber epoxy composite was 12 times higher than that of glass fiber epoxy composite. Espert et al. [35] investigated the impact of water absorption on the mechanical properties of cellulose fiber-reinforced composite. They discovered that increasing fiber content increases moisture absorption of the composite due to the hydrophilic nature of the natural fiber. Le duigou et al. [36] analyzed the effect of seawater on biodegradable flax reinforced polylactic acid (PLLA). The unreinforced PLLA had a saturated weight gain of 0.32%, while that of film-stacked specimens was 5.6%. It means that the weight gain of flax fiber at 20 °C was 17 times higher than that of PLLA.

All of the preceding discussion indicates that the natural fibers absorb a lot of moisture. Table 3 presents the moisture regained from various natural fibers. This low moisture resistance noticeably influences the mechanical performance of the composite. Many researchers have investigated the effect of moisture absorption of these natural fibers on the mechanical properties of the resultant composite. Le duigou et al. [36] reported that moisture absorption degrades the bonding between fiber and matrix and reduces tensile stiffness and strength. Yan et al. [5] analyzed the effect of various aging solutions on the mechanical properties of flax epoxy composite. The results show a significant reduction in the tensile and flexural properties of the composite. They noted a decrease of 22.6-31.1% in tensile strength, 24.0-36.4% in tensile modulus, and a reduction of 9.3-23.5% in flexural strength and 13.9-25.2% in flexural modulus. Dhakal et al. [37] found that flexural properties for both jute-reinforced composite and flax-reinforced composite decrease as the percentage moisture uptake increases. Compared with dry flax samples, the strength and modulus of the wet flax samples decreased by nearly 40 and 69%, respectively, while wet jute sample strength and modulus decreased by 60 and 80%, respectively. Moreover, water can have a plasticizaing effect and can plasticize the internal chemical structure [38].

Treatment of natural fibers with different chemicals reduces their moisture regain to a large extent [39]. Nayak et al. [40] treated areca sheath fiber with NaOH, sodium chlorite, permanganate, and benzoyl chloride. They observed that all chemical treatments reduced water absorption. However, when compared to other treatments, benzoyl chloride and sodium chlorite-treated fibers exhibited high moisture resistance. Dilfi et al. [30] investigated whether the combination of alkali and silane treatment reduces the moisture absorption rate of jute fiber epoxy composite. Teklu et al. [41] noted that sisal fiber with an alkali-treated surface and polyaniline coating, shows significant resistance to water uptake. The water absorption reduced from 20.9 to 9.8% after surface treatment, compared to untreated sisal fibers. Polyaniline-coated fibers had a much lower absorption (3.5%) as compared to alkalitreated uncoated fibers.

Polymer Matrix and Curing Agents

Epoxy Resin

Epoxy resins are special amid various thermosetting resins due to their unique properties: such as low creep, first-rate corrosion weather resistance, high-temperature performance, suitable electrical properties, lower shrinkage, and high availability of the resin in different forms (ranging from low viscous liquid to highly solid). Similarly, it requires minimum pressure for the product manufacturing. Their physical and mechanical properties can be adjusted to achieve extreme flexibility or high strength [42]. The resin can use several materials like glass, metal, wood, and natural fibers [43]. Due to these unique characteristics and valuable properties, epoxy resin is widely used in engineering composites, surface coatings, structural adhesives, and electrical laminates [44, 45].

Epoxy resin is a thermosetting polymer that contains at least one epoxide or oxirane group. The basis for all types of epoxies is the diglycidyl ether of bisphenol A (DGEBA). Nearly 90% of the world's epoxy resin development today is based on the reaction between bisphenol-A and epichlorohydrin in the of a simple catalyst, manufacturing DGEBA, as shown in Fig. 2.



Fig. 2 Synthesis of DGEBA

Curing Agents for Epoxy resin

Curing agents are also known as cross-linkers or hardeners. They are used to transform epoxy into thermoset networks. They can catalyze the curing reaction or initiate it through polyaddition and/or copolymerization with epoxy monomers. The structure of cross-linking agents considerably affects the properties of the cured products. Curing retailers are extremely important in the epoxy resin curing system because they are related to curing kinetics, reaction rate, curing degree, viscosity, and curing period, and thus play a significant role in determining the overall attributes of cured products. Active hydrogen compounds and their derivatives are the most common type of cross-linking agents. These hardeners entail amine, amide, hydroxyl, acid or acid group anhydride compounds. They generally react using polyaddition with epoxy resin to provide an amine, ether, or ester. Another type comprises of anionic and cationic initiators and is applied to catalyze epoxy resin homopolymerization. Molecules capable of creating anions such as tertiary amine, secondary amines, and metal alkoxides are efficient anionic initiators for epoxy resins. The third type of hardeners, also known as reactive cross-linkers generally, possess larger equivalent weights and crosslink with the 2nd hydroxyls of the epoxy resins or by self-condensation [46]. Table 2 presents some commonly used hardeners along with their chemical structures.

Natural Fiber Reinforced Epoxy Composites

Different physical and chemical approaches (Fig. 3) have been used for better fiber reinforcing, as mentioned in Sect. 2. Various researchers have analyzed and investigated these types of treatments [15, 47, 48].

Herein, we focus on improved treatment procedures, the use of bio-fillers, and hybrid approaches, all of which are increasingly being used to improve the quality of

Modification techniques

PHYSICAL METHODS	CHEMICAL MODIFICATION
• Corona treatment	• Alakali treatment
 Plasma treatment 	• Silane treatment
 Gamma Radiation 	• Enzyme treatment
	• Peroxide treatment
	• Stearic acid treatment
	• Etherification
	• Acetylation
	 Sodium chlorite treatment
	 Maleated coupling
	• Ozone treatments
	• Sodium chlorite treatment
	 Benzoylation
	• Grafting

Fig. 3 Physical and chemical treatments for modification of natural fibers

composites. The effect of hot alkali treatment on the tensile and bending properties of flax epoxy composite was investigated, in a typical study, as shown in Fig. 4. The study revealed that higher temperature fiber treatment accelerated the conversion of cellulose 1 to cellulose II, as obvious in XRD result in Fig. 4B. The treatment removed hemicellulose, some lignin, and other surface impurities such as wax and oil, creating cracks on fiber surface due to damage caused by alkali treatment (Fig. 4C). However, the removal of these impurities results in a cleaner fiber surface and a rougher topography, shown in Fig. 4C; the roughness increased with increasing treatment temperature, Fig. 4C(e). These rough fiber surfaces help in mechanical interlocking with the matrix; the cleaner fiber surface improves chemical interaction between fiber and matrix, resulting in increased fiber-matrix adhesion; and the appearance of cellulose II improved mechanical properties. The maximum





◄Fig. 4 A Process of alkali treatment. B XRD patterns of alkali treated flax fiber at different temperatures. C FE-SEM images of flax fiber a UF, b AFRT, c AF80, d AF120, e AF160. D Tensile properties of different temperature alkali treated fiber reinforced composite a tensile strength, b elastic modulus. E Bending properties of different temperature alkali treated fiber reinforced composite: a bending strength, b bending modulus

improvement in tensile strength (8.59%) and elastic modulus (50.4%) were obtained for composites treated at the highest temperature of 160 °C (the highest treatment temperature in this work), compared with the untreated composite Fig. 4D. The composite with fibers treated at 80 °C had 44.5% higher fracture energy. Similarly, bending strength and bending modulus improved 28.8% and 68.0%, respectively for composites with fiber treated at 160 °C (the highest treatment temperature used in this study) compared with untreated fiber composite [49].

Another technique to improve the properties is the inclusion of rigid/stiffer bioparticles in the epoxy matrix. Nevertheless, various factors such as the composition of the filler, the preparation process, improper dispersion of the filler in the matrix, filler matrix interactions, and filler orientation can all affect the mechanical properties of bio-fillers reinforced composites. In a recent study, the olive powder obtained from three different parts: olive tree small branch (OTS), olive tree big branch (OTB), and olive tree leaves, were reinforced in the epoxy matrix. Except for OTL composite, the tensile strength and modulus of the epoxy composites increased after with reinforcement. The flexural strength and modulus improved for composites with all three reinforcements (Fig. 5). The strength of the OTL fillerreinforced composite decreased due to the poor dispersion of OTL filler in the epoxy matrix. The SEM images (Fig. 5) corroborated these findings. Filler breakage was seen in OTS and OTB and filler pullout in OTL, showing improved interfacial interaction and stress transmission from matrix to filler, resulting in improved tensile and flexural properties for OTS and OTB. However, the impact strength of the composites decreased with the addition of olive filler. This reduction was due to the low strength of natural fiber compared to synthetic ones. With increasing immersion time, water absorption and thickness swelling increased for all composites [50].

The use of hybrid fibers or fillers is another promising technique that is becoming popular. The use of inorganic hard particles can minimize polymer chain mobility, redistribute stress, and improve composite stiffness. In recent research, basalt powder (2.5 to 10 wt%) was combined with six-layered basalt fabric in the epoxy matrix. The basalt powder composite with 2.5 wt% of basalt powder had the maximum tensile strength, elastic modulus, storage modulus, and glass transition temperature. It was due to the better dispersion of basalt powder in the epoxy matrix, as obvious

in SEM images (Fig. 6). The stiffness of the fillers caused a reduction in the elongation at break and impact strength of the reinforced composites [51]. Further advancements in natural fiber composites are covered in the following section.

Fiber Type

Epoxy, as a matrix, can be used to create a wide range of composites. They have broadly grouped into fiber reinforced plastic (FRP) composites, particulate composites, and nanocomposites [52]. The reinforcement of natural materials improves the physical and mechanical properties of the matrix. Natural fibers are divided into three main categories according to their origin; mineral, animal and plant fibers. Figure 7 depicts the various classifications of natural fibers [47, 53]. Animal fibers consist of proteins, and are not mostly favored for high performance except silk fiber. Silk fiber can give high strength, but its high cost and availability are the shortcomings. Woven silk fiber-reinforced composite exhibite far higher fracture strain abilities than glass fiber composites and are the appropriate selection to be used in places where high compliance is required [54]. Plant fibers are stronger and stiffer than animal fibers, making them ideal for structural applications [55, 56]. Plant fibers can be categorized as wood, bast, seed, leaf, and fruit fibers, depending on the plant portion from which they are extracted [57]. Cotton, jute, hemp, kenaf, flax, bamboo, coconut, and sisal are the commonly used plant fibers. The bast fibers such as flax, hemp, and jute are preferred for structural applications in composites because of their good mechanical properties [4]. Mineral fibers were outlawed in many countries due to their carcinogenic properties, hence they are less commonly used in high-performance technical applications [58]. The qualities of reinforced natural fibers have an impact on the performance of polymer composite. Table 3 demonstrates some characteristics of natural fibers [13–15, 54, 59]. Natural fibers properties depend on fiber type, harvesting time, growing conditions, extraction time, chemical composition and structure, treatment, and storage procedures.

Type of fiber is a critical parameter in defining the overall properties of the composites [60, 61]. Table 4 shows recent research work done on different natural fiber reinforced epoxy composites.

Fiber Volume Fraction

The demand for multifunctional materials in advanced engineering and the medical field is growing nowadays. The material properties and the structure (chemical and physical) both contribute to the multifunctionality. Materials having high strength, stiffness, fatigue, toughness, and low damping properties are classified as structural-functional materials, while others with electrical resistivity



Fig. 5 SEM images of the tensile fracture surface of epoxy reinforced with A OTS, B OTB, and C OTL composites. D Tensile strength and modulus of OTS, OTB, and OTL reinforced epoxy composites.

E Flexural strength and modulus of OTS, OTB, and OTL reinforced epoxy composites [50]

and thermal conductivity are classified as nonstructural functional materials. Generally, the main goal of industrial products is to minimize material weight and cost. Stiffness and strength, specific tensile stiffness and strength, and specific bending stiffness and strength are basic mechanical properties. Mechanical, thermal, and water absorption, all these properties are influenced by the amount of fiber in the composite [82]. Several natural fibers, especially bast fibers such as jute, flax, and hemp show good specific tensile stiffness than E-glass. As natural fibers have a lower density than E glass, therefore the reinforcement of natural fiber in polymer matrices at the same fiber content makes the final product almost 40% lighter than glass fiber reinforced composite [83-85]. Almari et al. [86] studied the effect of recycled cellulose fiber on the physical and mechanical properties of the epoxy composite. They demonstrated that bending strength, bending modulus, fracture toughness, and impact toughness increased with increasing fiber ratio. Furthermore, increasing the fiber ratio increases the fiber-matrix interaction, and the composites having higher fiber content have better interfacial bonding. They also discovered that composites with a high fiber content absorbed more water, with diminished interfacial bonds, and less fracture toughness, bending strength, and bending modulus. Fiore et al. [87] studied the effect of filler content and size on mechanical properties of Arundo donax fillers (ADFs) reinforced epoxy composites. They concluded that composites containing Arundo donax fillers display better tensile moduli, comparable to bending moduli, and lower strength characteristics as compared to pure resin. With the increase in particle size and content of ADFs, the storage modulus and loss modulus increased in the rubbery region. Islam et al. [88] studied hemp fiber reinforced epoxy composite with different fiber contents ranging from 40 to 65 wt%. They noted that untreated long fiber showed better strength at high fiber contents.



Fig. 6 A SEM images of composites a BF b 2.5BF c 5BF d 10BF magnification \times 1000. B The dynamic mechanical thermal properties (DMTA) curves of the composites. C Mechanical properties of composites [51]

Fiber Surface Modification

A variety of chemical and physical methods are available to amend the structure and properties of the fiber and increase the affinity between fiber and matrix. Fiore et al. [89] investigated the reinforcement of untreated and alkalitreated kenaf fiber in an epoxy matrix. When compared to neat resin both untreated and alkali-treated composites enhance mechanical properties. According to Mylsamy et al. [90], both length and alkali treatment of agave fiber epoxy composites show better adhesion for short fibers. Alkali treatment further increases fiber wetting and fiber-matrix interaction resulting in higher tensile compression, flexural, and impact strength. Yousif et al. [91] analyzed that the

Fig. 7 Classification of natural fibers



Table 3 Physical and
mechanical properties of
different natural fibers [13–15,
54, 59]

Fiber	Tensile strength (MPa)	Young modu- lus (GPa)	Specific modulus (GPa)	Elongation at break (%)	Density (g/cm ³)	Moisture regain (%)
Jute	200-460	20–55	14–39	1.5-1.8	1.34	12
Flax	510-910	50-70	34-48	2.7-3.2	1.45	12
Hemp	300-760	30-60	20-41	1.6	1.43	12
Kenaf	300-1200	22-60	_	1.6	1.30	17
Sisal	100-800	9–22	6-15	2.0-2.5	1.45	11
Banana	530	27–32	20-24	5.9	1.35	-
Abaca	14	41	_	3-10	1.52	14
Cotton	400-700	6–10	4-6.5	7–8	1.55	8.5
Coir	100-200	6	5.2	30	1.15	13
Bamboo	571	27	18		1.52	-
Bagasse	290	17	-	1.1	1.25	-
Silk	600	10	-	20	1.31	5–35

bending strength of untreated kenaf fiber-reinforced composite increases by 20% while alkali treatment of kenaf fiber further improves the bending strength by 36% due to interfacial bonding between fiber and epoxy matrix. Moreover, the porosity of the composite decreases with alkali treatment. Mahjoub et al. [92] studied the effects of changing NaOH ratios and treatment time on kenaf fiber epoxy composite. They found that 5% alkali treatment produces no tension on fiber texture and structure when compared to 10% and 15%, and is optimum for kenaf fiber treatment. Brodowsky et al. [18] while analyzing jute fiber-reinforced composite discovered that combining alkali and silane coupling agents improves the composite's adhesion strength and mechanical properties. Lu et al. [93] prepared untreated, alkali-treated, and silane-treated bamboo fibers reinforced epoxy composites. They concluded that alkali and silane treatment boosts composite tensile strength and elongation at break. They also discovered that NaOH treatment of the bamboo fiber increased the tensile strength and elongation at the break by 34% and 31%, respectively, compared to untreated cellulose reinforced composite. Likewise, the silane coupling agent also increases the tensile strength and elongation at the break by 71% and 53%, respectively. The effect of kenaf fiber on epoxy composite was studied by Azwa et al. [94], that the addition of untreated kenaf fibers increases thermal stability. But alkali treatment affects thermal stability, decreases degradation temperature of the composite, and produces less char than untreated fiber composite. They also observed that increasing composite exposure time only affects the decomposition rate up to 150 °C and has a minimum role in the decomposition of the composite. Anbukarasi et al. [95] discovered that NaOH-treated luffa fiber increases the thermal and mechanical properties of the epoxy composite. The composites containing particle fibers exhibited better water absorption. Hence, the particle fiber-reinforced composites can be employed in damp places like washroom doors, car

Table 4 Reinforcement of various natural fibers in the epoxy composite

Reinforcement	Characteristics	Year	Modifications	References
Natural mallow fiber	Ballistic efficiency	2017	Natural mallow fiber composite provides almost the same ballistic efficiency as Kevlar fiber composite, but mallow fiber is 20% lighter and 97% cheaper than Kevlar. This low density and reduce cost make mallow fiber a better choice for use in personal protection	[62]
Cornhusk fiber	Dynamic mechanical properties	2020	Cornhusk fiber improved the storage modulus and reduced the Tan δ of the epoxy composite	[63]
Banana fiber	Mechanical and thermal properties	2020	The study has concluded that banana fiber reinforcement up to 15% has increased the mechanical properties however, further increase of fiber above 15% has decreased the mechanical properties. The author had considered 15% banana fiber reinforcement in an epoxy matrix, the best according to their study	[64]
Hemp fiber	Mechanical properties	2020	30 vol% hemp fiber reinforced epoxy composite depicted good higher tensile and flexural properties compared to polyester composite	[65]
Coir fiber	Interfacial shear strength	2020	Liquid plasma treatment of coir fiber increased the interfacial shear strength of coir fiber epoxy composite	[66]
Flax and jute fabric	Mechanical and thermal properties	2020	Flax and jute fabric epoxy composites showed a good competition in thermal, mechanical, and dynamic mechanical properties related to glass composite	[67]
Flax fabric	Mechanical Properties	2021	Epoxy matrix was reinforced with four different 3D woven fabrics with vary- ing stuffer yarn densities. The surge in stuffer yarn density had highly upgraded the flexural and impact performance	[68]
Flax fiber	Mechanical properties	2021	The storage modulus of flax fiber (25% by volume) had shown an increase with frequency and reduction with tempera- ture. Soaking in the water had reduced the storage modulus and increased the loss factor	[69]
Hemp fiber	Mechanical and tribological properties	2021	Na_2CO_3 and H_2O_2 treatment of hemp fiber had resulted in an improvement in water resistance, tensile strength, and tensile modulus, wear, and frictional properties but, both treatments had decreased the impact strength of the composite	[70]
Lagenaria siceraria fiber	Mechanical properties	2021	Four different fiber lengths (3–9 mm) with five varying fiber contents (15– 35 wt%) were reinforced in an epoxy matrix. The best mechanical properties were found for 7 mm fiber length and 30 wt% alkali-treated fiber composites	[71]

Table 4 (continued)

Reinforcement	Characteristics	Year	Modifications	References
Fique, fique-cotton and moriche fibers	Mechanical properties	2021	The mechanical properties of the biocomposites were studied prepared with different vacuum pressures. Fique/epoxy and fique-cotton/epoxy composite had shown improvement in mechanical properties with an increase in vacuum value to 0.4. However further increase in vacuum value to 0.8 bar has shown a negative influence on mechanical properties	[72]
Aligned short harakeke fiber	Tensile properties	2016	The maximum tensile strength and young modulus of 136 MPa and 10.5 GPa, respectively, were noted for short-ori- ented fiber reinforced epoxy compos- ites produced with the dynamic sheet forming method	[73]
Sisal Fiber	Mechanical properties	2015	The flexural strength of 15 mm sisal fiber is increased by 25%, while the impact properties of 20 mm sisal fiber are greatly improved	[74]
Sisal fiber	Mechanical properties	2016	Results show that the ideal time for treat- ing sisal fiber is 120 h to get maximum mechanical properties by improving the interfacial bonding between fiber and matrix	[75]
Bamboo fiber	Tensile, impact strength, and water absorption	2019	Comparing with bulk natural bamboo, epoxy infiltrated bamboo composite improve 236% tensile strength and 136% impact strength. Furthermore, the epoxy infiltrated bamboo composite showed higher dimensional stability against moisture	[76]
Jute fiber	Surface topography, mechanical and thermomechanical properties, and durability test	2018	The combined alkali and silane treatment decreases the hydrophilic nature of the jute fiber and enhances the mechani- cal properties of the composite. SEM analyses of the fibers show a rough surface topography after various chemical treatments. Moisture exposure considerably lowers the mechanical and thermomechanical performance of the composite	[30]
Jute and basalt fabric	Tensile properties, water absorption, durability test	2018	Chemically treated jute fiber decreases water intake and increases the tensile strength of jute fabric composite. Comparing with basalt fiber composite all jute composites show high strength reduction by exposing them to different aging conditions	[77]
Silk fiber	Mechanical and thermal properties	2018	Silk/epoxy laminates exhibit similar ther- mal stability, glass transition tempera- ture, and thermomechanical properties as glass-reinforced epoxy composites. The specific flexural strength of silk composites is 23% greater than glass composites	[78]

Table 4 (continued)

Reinforcement	Characteristics	Year	Modifications	References
Bamboo fiber	Mechanical and thermal properties	2018	The findings reveal that both fiber con- tent and length increase the composite's bending modulus and fracture tough- ness monotonically. 6% alkali-treated bamboo fiber increase the thermal stability of the composite	[79]
Flax fiber	Mechanical properties	2018	10% sodium bicarbonate treatment increases fiber-matrix interaction. Hence, increasing tensile strength and modulus by 20% and 45% respectively	[80]
Hemp fiber	Mechanical properties	2018	The tensile modulus of silane-treated fiber composites is higher than that of untreated and alkali-treated fibers, and silane-treated fiber composite outperformed the alkali-treated fiber composite significantly. Also, 1% silane treatment is ideal for improving mechanical properties.	[81]

interiors, etc. Alkali-treated agave fiber composites have the lowest Tan δ at higher temperatures, according to Mylsamy et al. [90], making it a good polymer composite for high temperatures. Zhang [96] examined the microstructure and thermomechanical characteristics of natural bamboo fibers at various NaOH concentrations. They reported that as the NaOH concentration increases, so does the thermal stability. The thermal performance of unmodified and NaOHmodified coconut fiber reinforced epoxy composites was compared by Kumar et al. [97]. They perceived that composites with treated coconut fibers as a reinforcement had better thermal stability. Liu et al. [98] studied the impact of chemically treated abaca fiber on the transverse thermal conductivity of the composite. The results show better thermal conductivity for treated fiber-reinforced composite related to untreated fiber composite. Shih [99] examined the influence of silane treatment on the mechanical and thermal behavior of bamboo reinforced epoxy composites and concluded that silane treatment of bamboo fiber increases the thermal stability of the composite.

Bio-filler/Particles Reinforced Epoxy Composites

Bio-fillers derived from natural resources are increasingly being employed to improve the composite material performance while lowering the costs. Kranthi et al. [100] prepared pine wood dust reinforced epoxy composites. They concluded that pine powder holds good filler characteristics to improve the sliding wear performance of the matrix. Moreover, they discovered that specific wear rate depends on filler content, sliding velocity, and normal load. Biowaste - reinforced epoxy resin composites, such as fish scale reinforced epoxy resin composites, have low permeability and increased micro-hardness. These composites show the potential to be used in applications such as automobile seat covers, coal-taking pipes in power plants, conveyor belts, pipes carrying pulverized coal in power plants, turbine blades, and low-cost domestic items [101]. Shah et al. [102] fabricated acacia catechu particles reinforced epoxy composites and found an improvement of 14% and 94% in flexural and impact strength, respectively, with the insertion of 1 wt% acacia catechu particles. Rice husk reinforced epoxy composite was scrutinized by Rout et al. [103] and they observed that rice husk particles enhanced the sliding wear resistance of the polymer composite. Narendar et al. [104] noted that the chemical treatment increase crystallinity of coir pith resulting in improved compressive strength and storage modulus of the epoxy composite. Salasinska et al. [105] prepared a low-cost composite with enhanced mechanical and thermomechanical properties by reinforcing a large quantity of walnut shell powder into an epoxy matrix that could be used for low-performance application parts and applications. Shah et al. [106], on the other hand, investigated alkalitreated walnut shell reinforced composites and discovered that the addition of treated walnut shells increased thermal stability, storage modulus, and crystallinity index. In another research, Owuamanam et al. [107] compared untreated and stearic acid (SA) treated eggshell reinforced bio-epoxy composite. They came up with a conclusion that increase in filler content affects tensile strength, bending strength, and impact toughness. However, the bending modulus increases with the increase in filler content. Shah et al. [108] observed that stearic acid-treated eggshell particles reinforced epoxy composites showed reduced brittleness, increase thermal stability, improved stiffness, and greater glass transition temperature related to the pure matrix. Jabbar et al. [109] analyzed the reinforcement of NaOH treated pulverized jute into the epoxy matrix. They found that the addition of alkalitreated pulverized jute increases glass transition temperature, storage modulus and lowers the height of the tangent delta peak of the composite.

Natural Fibers/Fillers Based Hybrid Epoxy Composites

Natural fibers and materials derived from renewable resources are affordable and readily available in large quantities, making them perfect for the production of low-cost composites. Each material possesses unique properties and, considerable advantages and disadvantages based on its structure and properties. Fiber hybridization is one of the most common and rapidly expanding technology for improving composite qualities. The advantages of one fiber can balance the limitations of the other in a hybrid composite with different types of fillers. Hence, appropriate material design can yield a composite with a good balance in all attributes. Hybridization of bio-fillers with natural fibers is also a promising research direction [110]. Biofiber/filler hybrid reinforcements can modify material properties for the desired application and have the capacity to be used in every field like automotive, building materials, packaging, electronics, and chemical resistance [53]. At the same time, environmental concerns have drawn researchers' attention of researchers to manufacture high-performance composites with the combination of different kinds of biofibers and fillers for [111]. Considerable research has been done to reinforce natural fibers with other fillers to fabricate hybrid epoxy biocomposites. Some recent work done on natural materials hybrid epoxy composites is discussed below.

Hybridization of Natural Fiber Epoxy Composites

Hybridization of reinforcement fibers from natural resources with complementary characteristics, such as fiber/fiber or fiber/filler provides a set of properties that are difficult to achieve with a single material. Hybridization provides better properties as well as significant cost savings due to the use of low-priced raw materials. Sumesh et al. [112–114] investigated and optimized the various factors stabilizing mechanical and wear properties by combining different natural fibers in diverse ratios. They found that adding of 30 wt% of sisal fiber/pineapple fiber enhanced the tensile strength from 26.19 to 33.48 MPa, flexural strength from 67.79 to 73.07 MPa, and impact strength from 55.31 to 64.88 J/m [112]. In another study using the grey relational approach, they noticed that the optimum ratios for sisal and banana

fiber in the epoxy matrix were 20% sisal, 15% banana, and 5% NaOH, under 10 MPa pressure, and 100 °C temperature. In addition, there was a strong relation between experimental and predicted grey correlational grades for analyzing factors influencing mechanical properties [113]. Furthermore, they studied banana and coir fiber reinforced hybrid epoxy composites and had recorded optimized mechanical properties for 20% banana /15% coir/5% alkali treatment/16 MPa pressure and 100 °C temperature [114]. The hybridization of hemp and flax fiber with a total weight ratio of 30% in the epoxy matrix exhibited lower water absorption and higher mechanical properties with an increase in the percentage ratio of flax fiber. The storage and loss modulus was found to be maximum for 15% flax + 15% hemp epoxy composites [115]. The combination of flax and basalt fibers improved the impact properties of the flax fiber-reinforced composite [116]. The hybridization of bamboo/kenaf fiber with a 50:50 mixing ratio in epoxy matrix presented good dynamic mechanical and thermal properties [117]. Kenaf/areca fiber epoxy composite showed higher tensile, flexural, and hardness properties when kenaf fiber was used as an outer layer while improving the impact and compressive strength as a core material in the hybrid composite [118]. The addition of NaOH treated jute fiber increases the mechanical properties of sisal fiber reinforced epoxy composite [119]. Results demonstrate that adding about 50% sisal fiber to banana epoxy composite decreases moisture absorption and increased the mechanical performance of the composite [120]. The mechanical properties of hybrid composites were improved by adding woven jute fiber to pure oil palm empty fruit bunches (EFB) composites. Hybrid composite had higher tensile and bending properties than those of EFB composite, but lower than jute fabric composite [121]. The hybridization of Grewia optiva and Bauhinia vahlii fiber in epoxy resin improves the physical and mechanical properties of the composite [122]. The addition of lignin with hemp fiber can increase the impact, tensile, and bending strength. The addition of lignin up to 2.5% w/w augments both bending and tensile modulus [123]. The mechanical and thermal properties of jute-epoxy composite increase while hygroscopicity reduces with the inclusion of banana fiber, up to 50% [124]. The results obtained from dynamic mechanical analysis (DMA) and impact tests confirm that hybrid composite outperform non-hybrid composites in terms of residual bending strength and impact performance [125]. Basaltfiber reinforced hybrid composite can effectively upsurge the durability of biocomposite under salt fog environment conditions [126]. Gupta [127] concluded that the hybridization of natural materials is a practical approach to escalate the dynamic mechanical properties of the composite. Further, the chemical treatment of fibers shows improved mechanical properties of epoxy hybrid composites than generally used fiber composites [128]. Chaudhary et al. [129]

studied different combinations of natural fibers in epoxy polymer and observed better mechanical performance of hybrid composites. The hybrid composite containing jute, hemp, and flax fiber had the highest tensile strength, impact strength, and modulus. The maximum flexural strength for the hybrid composite was 86.6 MPa. Moreover, the chemical treatment increases the thermal stability of hybrid composites. Jute/curaua and jute composites presented better properties for the untreated conditions, while jute/sisal composite displayed better results under mixed treatment. However, the results of the untreated and mixed conditions jute/ramie composite depicted no significant difference [130]. Hybrid composites possess better micro-hardness and impact strength [131]. Tensile, bending strength, and modulus increase by increasing the flax fiber content [132].

Bio-fillers/Natural Fiber Reinforced Hybrid Epoxy Biocomposites

The use of organic and inorganic fillers in conjunction with natural fibers to make hybrid composites is becoming increasingly popular. The hybridization of bio-fillers with natural fiber is an effective research methodology to modify the properties of the composite. The combination of biofillers with natural fiber improves both mechanical and water absorption properties [133]. Stocchi et al. [134] prepared jute woven fabrics and fly ash particles reinforced hybrid composite. They determined that hybrid composite gave the best mechanical and fracture characteristics. Combining Azadirachta indica seed powder and spent Camellia Sinensis powder with jute fabric in an epoxy matrix increased the mechanical and thermal properties of the epoxy composite [135]. Calcined eggshell particles and alkali-treated sisal fiber reinforced hybrid epoxy composite can be used to make green composites because they have better insulation, water resistance, and mechanical properties than unreinforced epoxy [136]. Narendar et al. [104] prepared epoxy hybrid composite employing coir pith and nylon fabric. They concluded that the incorporation of chemically treated coir pith into epoxy resin improved its storage modulus. Furthermore, the combination of coir pith with nylon fabric improved the storage modulus. Chemical filler modification and hybridization improve the composite's mechanical properties and water resistance [137, 138]. Sumesh et al. [139] used banana fly ash, pineapple fly ash, and coir fly ash fillers (1–4 wt%) with 30 wt% of sisal (S)/pineapple (P) hybrid fiber composites as a reinforcement in an epoxy matrix. They had recorded tensile strength of 23.78-33.79 MPa by the substitution of Banana fly ash, pineapple fly ash, and coir fly ash fillers, compared to hybrid natural fiber composites (30 wt% sisal/pineapple), which showed a strength of 20.45 MPa. Similarly, by adding biowaste fillers, the impact and flexural properties were improved up to 21.77% and 22.11%.

The addition of calcium carbonate powder to a bagasse fiber reinforced epoxy composite improves the composite's bending and compression strength. Comparative analysis of the outcomes of chemically treated and untreated fibers reveals that the mechanical properties of chemically treated fibers reinforced hybrid composites are superior to those of untreated fiber-reinforced composites [140]. The bending, tensile, impact strength, and thermal stability of banana/flax, banana/kenaf, sisal/flax, and sisal/kenaf composites had significantly improved by different bagasse ash (BGA) content [141]. Hybrid composites with various ratios of eggshell particles with jute fiber epoxy matrix have been studied. The findings indicate that 10% eggshell powder composite gives higher impact, tensile and hardness properties [142]. Various mechanical and water absorption characteristics of groundnut and luffa fiber reinforced epoxy hybrid composites with different ratios of fiber content ranging from 10% to 50% have been studied. The results show that alkali-treated luffa fiber composite having 40% fiber content outperforms other fiber content composites. Luffa and groundnut reinforced hybrid composite exhibits higher mechanical properties than composite reinforced with only luffa fiber [143]. The mechanical and morphological properties of the composite improve significantly by adding palm oil nanofiller into kenaf fiber reinforced epoxy composite [144]. The addition of fly ash particles with natural cellulosic fiber improves the composite's tensile characteristics and surface quality. Fly ash particles behave as fillers, additives, surface treatment media, and cellulosic fiber as reinforcement elements to create a sustained smooth treatment of composite material. The produced hybrid composite is stronger than wood and plastic composites and can perform multiple functions [145].

Applications of Natural Fibers/Fillers Based Epoxy Composites

Automotive Industry

The use of natural fibers composites is mostly limited to nonstructural applications due to their brittle nature and are therefore primarily used in interior parts of the auto motives, door panels, packaging, cases, chairs, tables, and ironing boards. In 2012, the automobiles sector used more than 98% of the natural fiber-based composites developed in the European Union [146]. Rieter Company replaced glass fibers with abaca fiber and began commercial production of spare wheel pan covers for the Mercedes A-class car with reduced cost and weight, having a negligible effect on quality [147]. Rieter is also committed to developing the most advanced lightweight and eco-friendly sound packaging to enhance the recycling rate of individual components and minimize energy consumption and environmental effects. Daimler [148] manufactured sisal and flax fiber-reinforced epoxy composite with augmented mechanical properties and 20% lighter weight for the door panels of the Mercedes Benz E class. Natural cellulose fabric reinforced epoxy composites with an average strength higher than required for automotive threshold strength (25 MPa) have developed. Hence, bark fabric reinforced epoxy composites can be used for the interior parts of automobiles [149]. Mahdi et al. [150] studied date palm leaf fiber (DPLF) reinforced epoxy composite. They noted that date palm leaf fiber reinforced epoxy composites had a high energy absorbing capacity and proved to be a potent candidate as environmentally friendly materials for automotive applications. Jute fiber epoxy composites gave a better mechanical performance than polyester fiberreinforced composites and are preferable for application in the automobile industry [151]. Sumesh et al. [152] prepared pineapple/Flax epoxy composites with outstanding mechanical properties. The Pineapple/Flax natural fibers with peanut oil cake filler might be employed as a reinforcement material for making friction composites for brake pad applications. Calotropis gigantea fibers reinforced epoxy composites have demonstrated tribological performance in various engineering sectors, such as brake pads and brake discs [153].

Building Materials

Growing research in biocomposites demonstrates the significance of bio-fibers and fillers, which have applications in nearly every field. Biocomposites developed with natural fibers are broadly used in building components, transport sectors, furniture, packaging, marine, sports, and aerospace industries [56, 154]. Natural fiber- reinforced structural insulated panels (NSIPs) have shown better mechanical performance than oriented strand board structural insulated panels (OSB SIPs) and can be used to replace OSB SIPs [155]. Coir fibers are primarily employed to make a variety of floor decoration materials but the automotive industry also uses coir fiber for seat covers and to support seats [156]. Bagasse fiber composites can make medium-density fiberboard, that can be used in train cars [157, 158]. Natural fiber composite sheets outperform commonly used concrete and steel-made sheet files [159]. Likewise, natural fiber hybrid composites can be used specifically for cost-effective residential and interior parts [160]. Banana fiberreinforced composites are better alternatives and new materials used in low-cost household furniture, replacing wood, plastic, and conventional metallic, and non-metallic materials. Banana fiber-reinforced epoxy resin composites have been used in the design and manufacturing of multifunctional work tables. Woven fabric made from the natural banana fiber can be successfully reinforced in epoxy resin to develop a household stand for telephone [161]. Alkali-treated Teff straw fiber incorporated epoxy composites can be used for lightweight and semi-structural applications [162].

Medical Applications

Biocomposites materials also have a wide range of applications in the medical field. They can be used as an alternative to titanium, stainless steel, zirconium, and cobalt chrome, for fracture fixation in orthopedics [163]. Woven natural fiberreinforced epoxy materials are highly recommended in applications like patient positioning and support devices. Magnetic resonance imaging (MRI), X-ray computed tomography (CT) and radiotherapy results reveal that cotton and bamboo fiber reinforced epoxy composites have minimum impact on resulting images and are suitable for use in these applications [164]. Flax and ramie fiber reinforced epoxy composites provide comparable properties to the human femur and tibia. Moreover, when compared to other accessible materials for orthopedic implants, such as ceramic and metallic plates, these natural fiber-based composites are biocompatible, biostable, and ecofriendly. Therefore these hybrid composites are recommended in orthopedic implant applications [165].

Other Applications

Although natural materials-based epoxy composites are most commonly used in the automotive and construction industries, they have proven potential in almost any field. In a recent study, flax fiber-reinforced epoxy made pipes have demonstrated significantly higher internal pressure than the conventional pipe pressure range for domestic water distribution, and long-distance water transfer. It suggests a significant potential for application of flax fiber-incorporated epoxy pipes [166]. The fishing rod is made of materials that remove nanocellulose from vegetable roots [158]. The thermal decomposition property of coir particle composites is high enough to improve the conductivity of the sample, making it a preferable choice to be used in applications such as electrical and sensor devices [167]. Natural materials-based composites also have the ability to be used in musical instruments [168]. Natural fiber-based hybrid composites can be employed to prepare water and chemical tanks as they show chemical resistance [169, 170]. Epoxy composite reinforced with mallow fiber gives the same ballistic efficiency as Kevlar fiber and is used in military applications for personal ballistic protection [62].

Conclusion

This review emphasizes the importance of natural materials in high-performance composites over synthetic materials. The physical and chemical properties of these fibers, as well as the difficulty of making composite materials, are briefly discussed. Natural fibers show high capacity in producing new composites with high performance having a minimal environmental impact. However, more research is needed to eliminate variations in natural fiber performance attributes and improve the interface between natural fibers and polymer matrix. Furthermore, because natural fiber composites have low mechanical properties compared to synthetic fibers, they are used for nonstructural applications. Hence, for structural applications, hybridization of more than one material from natural resources in polymer composites can increases the mechanical and thermomechanical properties that meet the required standards of the civil engineering materials field.

Furthermore, the physical, thermal, and mechanical properties of natural fiber/fillers reinforced epoxy composite/hybrid composite are ideal for the fabrication of highperformance engineering materials. The reinforcement of natural fibers and fillers is an impressive way to improve the mechanical properties of epoxy composites. Bio-fillers reinforced epoxy composites can be a better substitute synthetic ones in engineering applications requiring low to medium structural strength.

To overcome the variation in fibers advanced retting techniques should be applied to produce high-quality fibers and a better classification of the fibers; in terms of their diameter and length based on their end-use to minimize irregularity. Moreover, fiber surface sizing with different nanomaterials can enhance the interfacial properties of the composites and needs further exploration. Similarly, a combination of various materials (dual coating) on the surface of natural fibers can improve the interfacial adhesion of the composite and hence can provide better strength and toughness, allowing it to be used in structural applications. Furthermore, adding hybrid materials and surface coating with functional materials can improve flame retardancy, electromagnetic shielding performance, water-resistance, tribology, and thermal properties and could serve as a substitute for conventional materials. In addition, different natural fibers, which have yet to be studied as a reinforcement, must also be taken into account.

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Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Fazal Maula Khan received his master degree (2020) in Materials Science and Engineering from Harbin Engineering University, China. Now he is a Ph.D. student in Materials Processing Engineering under the supervision of Prof. Yan Zhao at the school of Materials Science and Engineering, Beihang University, China. His research interests are high performance fiber reinforced polymer composites, functional and nanocomposites.

Ahmer Hussain Shah is an Associate Professor & chair in the department of Textile Engineering, Balochistan University of Information Technology, Engineering and Management Sciences (BUITEMS), Pakistan. He obtained his Ph. D. degree (2019) in Material Sciences and Engineering from Harbin Engineering University, China. His research interests are biomaterials and fiber functionalization, fiber reinforced composites, epoxy based plastics, high performance polybenzoxazine com-

posites, nano material synthesis, silver, titanium, carbon, graphene, selenium and Mxene nano particles for medicinal and dye degradation properties.



Shuo Wang received his master degree (2020) in Chemical Engineering and Technology from Harbin Engineering University, China. Now he is working as purchasing supervisor in Zhongxing Telecommunication Equipment Corporation (ZTE), Shenzhen, China.









Shah Mehmood received his master degree (2021) in Materials Science and Engineering from Harbin Engineering University, China. Now he is pursuing his Ph.D. degree in Materials Science and Engineering at University of Science and Technology of China, and undertaking a Ph.D. program at Suzhou Institute of Nano-Tech and Nano-Bionics, Chinese Academy of Sciences. He is working on the construction and applications of nanocomposites hydrogels.

Jun Wang received her Ph. D. in 2010 from Harbin Engineering University, China, majoring in Materials' Science. She is a Professor in the College of Materials Science and Chemical Engineering, Harbin Engineering University, China. Her research interest includes the synthesis, curing kinetics, and properties of highperformance resins and related composites.

Wenbin Liu is a Professor in the college of Materials Science and Chemical Engineering at Harbin Engineering University, China. From 1987 to 2002, he worked in Institute of Petrochemistry Heilongjiang Academy of Sciences. Now he is a Ph. D. supervisor in Materials Science and Engineering. His research interest includes synthesis, polymerization mechanism, and structureproperty relationship of highperformance polymers and polymer matrix composites.

Xiaodong Xu is an associate professor in the College of Materials Science and Chemical Engineering at Harbin Engineering University. She received her Ph. D. degree (2005) in Polymer Chemistry and Physics under Prof. Jinghua Yin's mentorship at Changchun Institute of Applied Chemistry, Chinese Academy of Sciences (CAS). She joined Harbin Engineering University in 2005. Her main research interests focus on advanced polymer materials and polymer composites.