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Review

Recent Trends in Treatment and Fabrication of Plant-Based Fiber-Reinforced Epoxy Composite: A Review

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Abstract: Natural fiber (NF) is one of the many resources that nature has provided. NFs decompose quickly and are biodegradable, renewable, and cost-effective. It may be scavenged from a variety of plant and animal sources. They are employed as reinforcing materials in polymers for NF composite development. Because of its environmental friendliness and long-term survivability, NF is growing in appeal among academics and researchers for usage in polymer composites. This study aims to offer a thorough evaluation of the most suitable and widely utilized natural fiber-reinforced polymer composites (NFPCs), along with their manufacture, processing, and applications. It also defines several external treatments of NF and their influence on the characteristics of NFPCs. The characteristics of NFPCs are affected by fiber supply, fiber type, and fiber structure. Numerous physical and chemical treatments were tested to see how they affected the thermal and strength properties of natural fiber-reinforced thermoplastic and thermosetting composites. Several polymer composite fabrication techniques were also studied. NFPCs have several disadvantages, notably low fire protection, poor strength properties, and greater moisture absorption, which have prevented their application. It is shown how NFPCs are employed in a variety of industries, particularly automotive and research industries. The review discovered that intentionally changing the regular fiber enhanced the thermochemical and physico-mechanical properties of the NFPCs by means of improving the grip between the fiber surface and the polymer framework. This study aims to provide important and fundamental facts on NF and their composites, which will aid in new investigations, the creation of a creative framework for polymer composite types, and the achievement of Sustainable Development Goals.

Keywords: agro-waste; epoxy; fabrication techniques; natural fiber; polymer composite; surface modification

1. Introduction

Though the notion of developing bio-friendly materials appeals to both industrialists and environmentalists, when mechanical qualities are examined, natural fiber-reinforced polymer composites (NFPCs) fall well short of glass fiber composites [1]. Another area of composite science study has been inspired by the need to enhance the mechanical and actual characteristics of NFRCs and has identified poor holding between natural plant strands and the polymer framework as the primary cause of poorer mechanical property estimates [1]. Special attention is given to treatments for fiber surfaces which render them suitable for the structure, hence increasing the strength of the composite [2,3].

NF essentially consists of strands that are neither designed nor created by humans. They are derived from both animals and plants [4]. In recent decades, there has been a lot of interest in using NF produced from mutually nonrenewable and renewable assets, such as flax, jute, palm oil, and sisal, and to make composite materials [5]. Plants that produce cellulose filaments include jute, seed filaments, hemp, ramie, kenaf, flax, leaf filaments (sisal, abaca, and pineapple), reed filaments (wheat, rice, and corn), grass, primary components (hemp, jute, and kenaf), and any remaining variants (wood and roots) [6]. Table 1 indicates evolution of publication per year, Table 2 depicts the globe's extremely common and efficiently feasible NF in addition to worldwide manufacture [7]. Due to modern composites being frequently constructed of synthetic polymers, several environmentalists have recently criticized their usage. There are several reasons why it affects the environment [8]; to begin with, synthetic polymer manufacture is dependent on petrochemical raw ingredients, which not only rely on diminishing fossil fuels but are also non-biodegradable. Furthermore, the manufacture of this polymer generates a significant quantity of carbon dioxide. Because synthetic polymers are not biodegradable, their disposal is hazardous to both marine life and animal ecosystems [8,9].

Table 1. Evolution of published articles on natural fiber per year.

Year	Number of Publication	Percentage
2012	814	5%
2013	898	5%
2014	1020	6%
2015	920	5%
2016	1349	8%
2017	1522	9%
2018	1750	10%
2019	1790	11%
2020	1989	12%
2021	2130	13%
2022	2494	15%
2023	203	1%

Natural fiber has more desirable qualities and advantages over engineered filaments, such as comparably low weight, low price, fewer impacts on handling equipment, excellent comparative mechanical characteristics such as malleable yield strength and flexural modulus, expanded exterior clean of composite formed pieces, sustainability, and overflow [10], combining processing flexibility, biodegradability, and resulting NFPCs with large specific strength and stiffness [11–13]. Carbon nanotubes, cellulose nanocrystals, and synthetic graphite fillers are attached to the pattern to increase the composite strength attributes. These filler compounds improved composites' tensile, flexural, and fracture toughness qualities, as well as their electrical conductivity [14–16]. Each year, it is predicted that about 16 million automobiles are produced in the western part of the European Union alone, utilizing about 80k to 160k tons of natural fiber [17]. However, there are severe property deficiencies in natural fiber. The structure of NFs (lignin, cellulose, gelatin, waxy components, and hemicelluloses) interacts with environmental water penetration to form weak linkages between polymers and fibers. Moreover, due to the material designs of the filaments and the framework change, combinations between polymers and natural fibers are anticipated to be complicated. These are the reasons for inadequate pressure transmission when the created composites come into contact, hence regular fiber replacements with particular treatments are absolutely required [7,18,19]. Depending upon the application, this knowledge may be put to use in a wide range of ways.

Table 1 indicates that research on this particular subject had eight hundred and fourteen (814) as the lowest number of articles and a maximum number of articles in 2022 of two thousand four hundred and ninety-four (2494). It could also be observed that the year 2023 has the fewest articles as the year has just started, but has two hundred and three (203) articles already.

As such, this review article brings together information on contemporary details of surface treatment (chemical and physical), manufacturing processes, fiber/matrix selection and application of NF, polymer composites, factors affecting properties of the composite, diverse kinds of methods applied for polymer composites, and information on NFs and the impacts of their use in composites systems. With this review, detailed knowledge on the impacts of natural fiber polymer composites can be presented (NFPC). Different types of fibers for different applications are shown in Figure 1.

Figure 1. Diverse applications for hybrid natural/synthetic reinforced fiber polymer matrix. (**a**) Sugar palm fiber (**b**) Pineapple fiber (**c**) Hemp fiber (**d**) Corn fiber (**e**) Jute fiber (**f**) Flax fiber (**g**) Coir fiber (**h**) Kenaf Fiber (**i**) EFB fiber.

Table 2 summarized the strength the range properties of different natural fiber.

Table 2. Strength properties of different NF [7,20–45].

Fiber Type	Density (kg/m^3)	Young's Modules (GPa)	Tensile Strength (MPa)	Tensile Strain at Break	Ref.
Abaca	1.5	12	400	3.0–10.0	[7]
Areca	0.7–0.8	1.12–3.15	147–322	10.23–13.15	[20–22]
Bagasse	1.2–1.25	17–27.1	20–290	1.0–1.1	[23–25]
Banana	1.3–1.35	17.0–33.8	345–520	1.7–53	[26]
Bamboo	1.5	27	575	3.0	[27]
Coconut	1.15–1.46	2.8–6	95–230	15–51.4	[28]
Cotton	1.51–1.60	5.5–18	287–800	3.0–10.0	[29]
Coir	1.20–1.25	4.0–6.0	175–220	15.0–25.0	[30]
Feather	1.3	5.0–25.0	100–1500	15.0–30.0	[31]
Flax	1.40–1.50	18.0–80.0	345–1830	1.20–3.20	[32]
Harakeke	1.3	14.0–33.0	440–990	4.20–5.80	[33]
Hemp	1.48–1.50	70	345–1110	1.6	[34]
Henequen	1.4	*	430–580	3–4.7	[35]
Jute	1.5–1.5	10.0–55.0	200–800	1.5–1.8	[36]
Kenaf (bast)	1.22–1.40	22–60	295–1191	1.6–6.9	[37]
Kenaf (core)	0.21	*		*	[38]
Palm	0.7	80–2480		17–25	[39]
Pineapple	1.5	170–1672		1.0–3.0	[40]
Ramie	1.5	220–938		2.0–3.80	[41]
Silk	*	20–25		7.32–11.22	[42,43]
Sisal	1.33–1.5	400–855		2.0–3.0	[44]
Wool	1.3	50–315		13.2–35.0	[45]

* = Not applicable.

2. Fiber Type/Selection

Fiber is often classified as a plant, animal, or mineral by its origin. Although animal strands are mostly constituted of protein, all plant filaments are primarily composed of cellulose. Although mineral-based NF may be found in the asbestos group of materials and was previously often used in composites [46], they are now prohibited because of safety concerns (known carcinogens via respiration) and are banned across many nations. Researchers and scientists [34,38,40] are also focusing more on using non-depletable resources to protect the environment, eliminate waste, and avoid using non-renewable resources [46]. Numerous studies on natural fibers (NF) that are adequate substitutes for mock materials (carbon fibers and glass) in complex purposes have been conducted, including empty fruit bunch (OPEFB), sisal, palm oil ramie, bamboo, hemp, kenaf, vakka, date palm, jute, coir, and flax [47–50]. There has been a significant amount of interest in using NF produced from mutually renewable and non-renewable assets, such as sisal, palm oil, jute, and flax, to produce composite materials in recent years [51].

Bast strands (kenaf, hemp, flax, ramie, and jute), leaf filaments (pineapple, abaca, and sisal), seed strands (coir, kapok, and cotton), main components (jute, kenaf, and hemp), grass and reed filaments (corn, wheat, and rice), and any remaining varieties are all plants that produce cellulose filaments (wood and roots) [52]. NFs, for instance, pineapple leaf fiber, banana, sisal, coconut coir, hemp, and jute, have been employed in complex products in the past. Because plant stem fibers come with advanced strength properties and are more valuable, researchers have been investing in the development of novel types of stem fibers as well as the fabrication of their composites in recent years. In addition to cellulosic fibers, several researchers are pioneering the utilization of natural particles as reinforcements [41,53,54]. However, integrating both will provide a broad scope and research opportunity for the creation of novel NF and particles in the production of polymer composites [41,53,54]. Figure 2 shows the classification of different natural fibers.

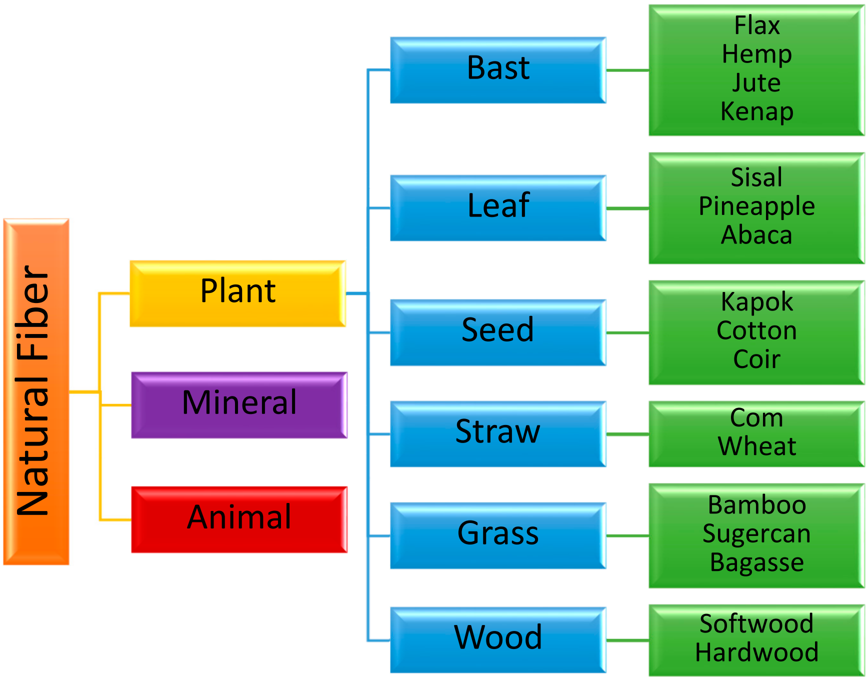


Figure 2. Schematic representation of natural fibers.

Modern polymer composites frequently use regular fiber as powder fillers to enhance the optimal characteristics of the composite above the original virgin or pure gum. This is undertaken in three different configurations to account for different proportions [53,55,56]. The second approach includes using short fiber and randomly inserting it in a predefined proportion of volume content. The third technique includes the use of continuous bio-fiber in polymer composites [53,55]. In general, higher-performance plant fibers may achieve substantially greater strength and stiffness than commonly accessible animal fibers. Silk is an exception to this rule since it is extremely strong but also relatively pricy, less solid, and far less frequently available. Thus, plant-based strands are frequently eligible to be utilized in composites with essential criteria, and this audit focuses on them. Additionally, plant fiber might be created and harvested quickly in a number of nations. Table 2 lists the features of a particular NF. While it has been demonstrated that among cellulose-based normal filaments, flax, hemp kenaf (bast), and pineapple strands have the finest elastic properties and Young's moduli, it should be kept in consideration that there is a significant lot of variation in the literature [55,57]. Geology related to fiber accessibility frequently has a significant impact on fiber selection. Genotypes with greater cellulose content and cellulose microfibrils located closer to the fiber heading typically produce better results. These strands, for example, kenaf, jute, hemp, flax, and ramie, come with greater primary needs for maintaining the plant's stem.

Natural fiber properties vary greatly based on synthetic synthesis and structure, which are related to fiber type, development circumstances, collection period, extraction interaction, treatment, and capacity systems. Strength is thought to deteriorate by 15% more than 5 days beyond the optimal collect time [55], while hand-extracted flax fibers are 20% stronger than mechanically extracted flax fibers [40]. Young's modulus reduces when moisture content decreases, while strength normally increases as moisture content lowers and decreases as temperature rises [19,41,58,59]. Given that the strands are often more grounded and stiffer than the matrix, it seems to make sense that with increases in fiber content, the firmness and strength of the composite improved. Maintaining an acceptable fiber/network interfacial strength, however, is required for this, and with particularly hydrophobic materials such as polypropylene (PP), strength might decline as fiber concentration grows if coupling experts or another interfacial designing approach is utilized. Despite this, elastic modulus grows with fiber concentration, but more gradually than when the connecting point is not regulated [60].

High fiber volume fractions are a problem not just for short-term composite qualities, but also for the possibility of higher water absorption, which can contribute to the deterioration of longer-term properties [61]. In comparison to composites with a grain capacity of 0.3, which consumed just 7% water and attained immersion in a comparable period of time, hemp fiber-supported PP composites with a grain capacity of 0.69 retained about 53.1% of the humidity and did not reach immersion until later at nineteen days [61]. Atiqah discovered that composites with nine weight percent of natural fiber with honeycomb had the maximum effect hardness and strength, reaching 58.088 HB and 98.1 J/m, respectively, in research on the hardness characteristics and impact of natural fiber honeycomb with epoxy composites reinforced. These findings show that beeswax honeycomb has the potential to be applied as a filling in reinforced composite [62]. The span of fibers is an essential component in establishing composite strength characteristics and must be borne in mind when computing its aspect ratio (length/diameter). The mechanical characteristics of composites are one of the primary elements influencing how a fiber’s ratio (L/D) impacts its length. Shear at the fiber/matrix contacts in a little composite transmits fiber tensile force on or after the matrix into the fiber [63]. Tensile stress is zero just at the extremities of the fiber and rises across its length; hence, a fiber must be longer than a critical length (Lc) to be destroyed under composite tensile loading [64]. The fiber might have hypothetically sustained half the load of a constant fiber at the identical composite strain at the critical size, right before rupture. To enable efficient composite reinforcement, the fiber size should be greater than the critical fiber span, allowing the bulk of the fiber to be loaded as if it were a continuous fiber [65].

Natural fiber has attracted significant attention in a widespread series of industries because of its superior advantages over synthetic fibers in terms of less destruction to processing facilities, relatively low expensiveness, relatively low mass, and good relative mechanical features, for example, renewable resources, tensile and flexural modulus, the enhanced surface quality of composite molded products, and abundance in nature [7,10,66]. Because the fibers are collected from various portions of the plant, they are categorized as leaves, stems, bast, fruits, and stalks. Coir fibers derived from cocus Nucifera plant fruit separate the hard, outer shell of a coconut and the internal crust. The plant’s stem outer cell layers are collected and used to remove bast fibers. Jute, vines, ramie, flax, and rattan are rare examples of fibers. Leaf fibers are fibers that are taken from the leaf cells of plants such as bananas, pineapples, and other similar plants. Table 3 provides a list of the most popular NF and particles [33].

2.1. Factors Affecting Properties of the Composite

NFs suffer from a number of problems that harm composite characteristics. These fibers have diverse chemical components that make up their structure, and as a result, they process with varied degrees of reactivity to the matrix and environment. The subsequent primary difficulties must be tackled while developing composite materials.

2.1.1. Loading, Orientation, and Length of the Fiber

The features of fiber-reinforced polymer composites are influenced by their volume or loading percentage, their dispersion, the size of the fibers, and their orientation within the matrix. When a force is applied to the matrix, a shear force is transferred at the interface over the full length of the fiber as well as at the extremities. The crucial fiber size, the orientation and direction of the fibers with respect to one another, and the stability of the fiber–matrix junctions all influence how much load is delivered toward the fiber. Three types of fiber reinforcement can be formed depending on how the fiber is orientated at the matrix. Longitudinally oriented fibrous composites have better tensile strength but poorer compressive strength due to fiber buckling. Second, fiber may be able to bear very little tensile stress in a transverse direction—less than the matrix’s strength. Last but not least, due to the dispersion, direction, and complexity of the load distribution towards the fiber matrix edge, it is more difficult to predict the strength characteristics of casually positioned short fiber composites. Aspect ratio, dispersion, and fiber orientation are a few examples of factors that may be changed to significantly improve the qualities of composite materials [28,33,37–39].

2.1.2. Structure of Plant Fibers

Cellulose has mutually amorphous and crystalline parts in its structure. Figure 3 depicts a schematic illustration of the secondary layer of the fiber cell block, which contains both amorphous and crystalline areas. Powerful hydrogen intramolecular bonds are created between large molecules and crystallite cellulose. Due to the cellulose blocks formed by the crystalline region's compactness, chemical penetration is challenging. On the other hand, the amorphous zone easily absorbs colors and resins. Plant fibers have hydroxyl groups in their structures, which makes them very polar and hydrophilic. NF must be chemically treated to improve the crystalline area, wipe away impurities (waxy compounds), and erase hydrophilic hydroxyl groups. To alter the chemistry of fibers, surface treatments (physical, chemical, and biological) are frequently applied [47].

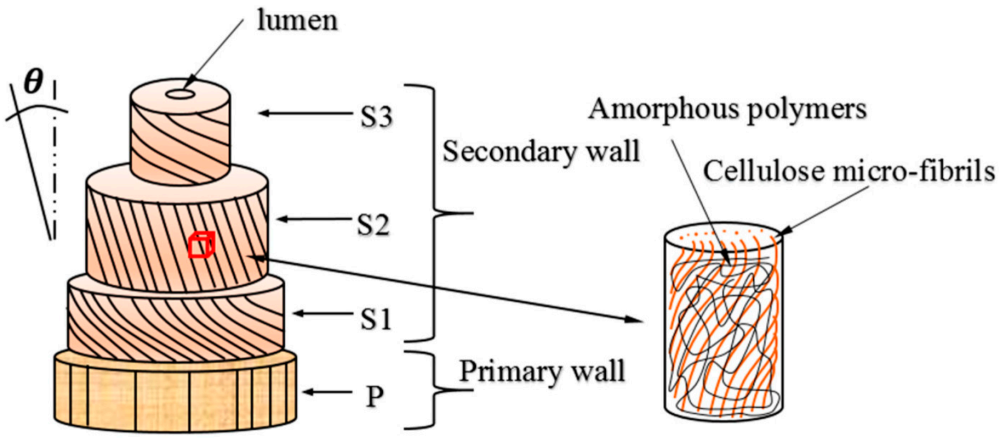


Figure 3. A single fiber has primary and secondary layers, with the intermediate layer consisting of S1, S2, and S3 layers. A lesser section of the S2 sheet is enlarged, revealing celluloid micro-fibrils scattered atop an unstructured polymeric substrate [67].

2.1.3. Fiber Thermal Stability

The degradation of natural fiber starts at about 240 °C. Temperature is a factor in the behavior of the fiber's structural constituents, including cellulose, hemicelluloses, lignin, and others. While hemicelluloses and cellulosic components begin to decompose at greater heat levels, lignin tends to break down at around 200 °C. By chemically removing a specified quantity of lignin and hemicelluloses from the fiber, thermal steadiness can be increased. Natural fiber composites are becoming increasingly popular in composite manufacture (injection molding, extrusion, or curing) and in-maintenance materials, raising severe issues regarding natural fiber deterioration [68].

2.1.4. Voids Existence

During the introduction of fiber into the matrix, volatile substances or air may become trapped in the material. Microvoids may form in the composite after curing as a result of the fiber gap between both the laminate and the resin-rich areas, lowering the strength properties of the composites. Vacuum creation can also be caused by the pace of cure and cooling of the resin [68–71]. Higher void content (more than 20.1% by volume) results in weaker fatigue resistance, a larger affinity for the diffusion of water, and greater variance (scatter) in mechanical characteristics [72,73].

2.1.5. Fiber Moisture Absorption

Water is immersed by the hydrophilic lignocellulosic fibers. The plant fiber cell wall has a considerable degree of hydrogen bonding among the macromolecules (hydroxyl groups -OH). When the fiber is exposed to humidity from the environment, the hydrogen link splits and the hydroxyl forms new hydrogen bonds with the molecules of water. Water penetrates the material most commonly through the fiber's cross-section [74,75]. As a result, the matrix expands whenever hydrophilic fiber is replaced with a hydrophobic resin. Composite materials suffer from weak mechanical characteristics, poor fiber-to-matrix bonding, matrix cracking, and dimensional variability [74,75]. As a result, before creating composites, it is required to remove moisture from fibers. Moisture absorption in NF can be minimized by removing hydrophilic hydroxyl linkages on the surface of fiber using various chemical cures [76,77]. Figure 4 is showing the factors that influence the composite performance, Table 3 is showing the different fiber types and their scientific name.

Table 3. Fiber types and their scientific name [6–28].

	Type of Fiber	Common Name	Family Name	Ref.
1	Bast Fibers	Hemp	Cannabis-sativa	[6]
		Jute	Corchorus-capsularis	[7]
		Flax	Linum-usitatissimum	[8]
		Kenaf	Hibiscus-cannabinus	[9]
		Roselle	Hibiscus-sabdariffa	[10]
		Ramie	Boehmeria-nivea	[11]
		Rattan	Hordeum-vulgare	[12]
2	Leaf Fibers	Abaka	Musa textilis	[13–16]
		Henequen	Agave -fourcroydes	[17]
		Pineapple	Ananas-comosus	[18–20]
		Banana	Musa mannii	[21]
3	Seed Fibers	Kapok	Ceibapentandra	[22]
		Cotton	Gossypiumarboreum	[23]
4	Stalk Fibers	Rice	Oryzasativa	[24]
		Bamboo	Bambuseae	[25]
5	Fruit Fibers	Coir	Cocosnucifera	[26]
		Tamarind	Tamarindusindica	[27]
6	Stem fiber	Sisal	Agave sisalana	[28]
		Crown	Calotropisgigantea	[19]
7	Particulates	Groundnut shell	Arachishypogaea	[21]
		Seashell	Cypraecassisrufa	[27]
		Jack fruit	Artocarpusheterophyllus	[28]

2.2. Matrix Selection (Epoxy)

Epoxy resin vinyl esters, epoxy, and polyesters are popular thermosetting matrix materials utilized in automotive, infrastructure, aerospace, marine, chemical, and electrical applications. When compared to polyester, epoxy has superior mechanical qualities. Vinyl ester adheres well to a wide range of fibers and other substrates. However, in most engineering applications, epoxy is employed extremely efficiently as an adhesive and as a laminating resin. When employed in polymer composites, it provides good moisture barrier properties. It bonds extraordinarily effectively to fibers, allowing it to utilize and create fiber-reinforced polymer composites. Small amounts of a reactive curing agent are introduced shortly before putting the fibers into the liquid mix to start the polymerization reaction that turns the liquid resin into a solid resin [33]. An amine-based hardener (HY 951), for example, is recommended by the supplier for the manufacturing of composites at a 9:1 mixing ratio. M/S Covai Seenu & Company, Coimbatore, Tamil Nadu, India, supplied the epoxy resin (LY 556) and hardener [76–80] that were used in this study [33].

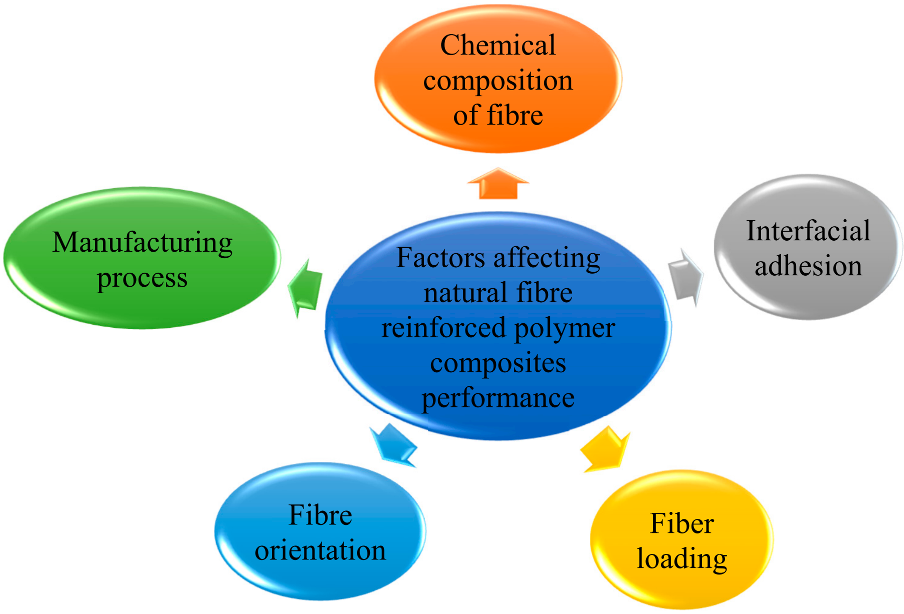


Figure 4. Factors that influence the composite performance.

2.3. Matrix Selection

Flexural, impact mechanical characteristics, and tensile properties of complex materials are all significantly connected to the fiber interface, with inadequate grip between these components resulting in poor load distribution [78]. As a result, selecting the optimal fiber/matrix blend is critical for achieving acceptable thermal and mechanical characteristics in composite materials. The matrix maintains the reinforcing fibers in the proper place, guards against mechanical and chemical damage, and makes sure that the load among the fibers transfers efficiently [79]. The most often utilized polymeric matrix in the fabrication of hybrid NF composites are those that are lightweight and can be made at low temperatures. Figure 5 is showing various types of polymeric matrices.

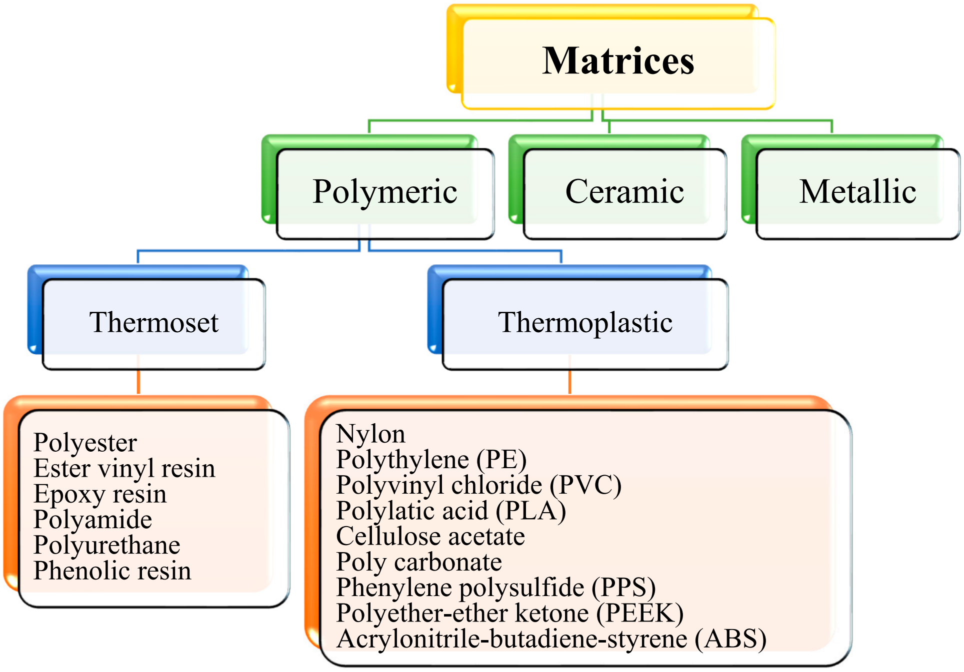


Figure 5. Various types of polymeric matrices.

Without a matrix, a fiber-reinforced composite would be lacking. It serves as a barrier against extreme conditions, guards the surface of the fiber, reduces wear on mechanical components, and transfers weight to the fibers. Modern NFCs most frequently employ polymeric matrices because they are lightweight and can be generated at low point temperatures [80]. In matrices such as NF, both thermoset and thermoplastic polymers have been used [80]. The temperature at which NF degrades is a constraint on matrix selection. The bulk of the NFs used as strengthening in NF composites are thermally insecure beyond 200 °C; however, in certain conditions, they may be handled for a short time at greater temperatures. As a result of this limitation, only thermosets and thermoplastics could possibly be used as matrices [81].

It should be stressed, however, that the use of the aforementioned thermoplastics is substantially more than that of any other regularly used thermoplastic matrices in the plastics industry. In reality, PP and PE are the most frequently used thermoplastic resin matrices for NFCs. Phenol formaldehyde, VE resins, epoxy resin, and unsaturated polyester (UP) are the most common thermosets. Thermoplastics could be softened and stiffened repeatedly by heating and cooling, which gives them the ability to be among the most easily reused elements, making them the preferred choice in current innovative deployment. However, thermosets typically realize fiber qualities more effectively. Investigations on the usage of bioderived matrices as opposed to petroleum-based matrices have been conducted [82]. In terms of mechanical qualities, PLA is clearly the victor because it offers higher strength and stiffness with NF than PP [82].

3. Techniques Used for Polymer Composite

3.1. Hand/Wet Lay Up

The mold is initially filled with fibers. It is possible to weave, sew, or bind fibers. In order to impregnate the resins, rollers, brushes, or other impregnators are used. The laminates created using this technique are then dried in a typical atmosphere. Even when combining fiber and resins such as vinyl ester, epoxy, and polyester, there are no such constraints on general materials. The method is simple to use, inexpensive, and performed at room temperature. This method may be used for any matrix and fiber combination. In comparison to another method, this one employ fibers with greater fiber content and longer fibers. Since this is a handcrafted process, more care must be taken. In this method, less viscous resins are utilized to ease labor. Because the quality of the product is determined by the worker, highly skilled workers are necessary. Homogeneous resin dispersion in the fiber cannot result in voids. This method is excellent for producing wind turbine blades, boats, and other similar items [83].

3.2. Lay-Up Spray

The fiber is cut by hand, filled with armament, and the mold is sprayed with resin. The products are believed to heal under normal atmospheric conditions. Polyester resins that have been glass roved are appropriate for this technique. This technique of production is unsuitable for larger structural pieces. It is also challenging to control the fiber volume percentage and thickness. The quality of a product is determined by the abilities of the workers. Because of the open nature of this approach, styrene emission occurs. It only has a decent surface polish on one side. Spray layup fabrication is not appropriate for items requiring great dimensional precision. As with hand layup, a low-viscosity resin is desirable. Baths, shower trays, a few small dinghies, and vehicle fairings are examples of light-stacked basic boards [37,60].

3.3. Filament Winding

This is a fully automated invention that is typically used to produce flexible fiber components or structures. Using this method, one may create empty spherical pieces. Mandrel rotation rate and fiber handling tools affect fiber direction. Inside the oven, the wound gets repaired. Polyester, vinyl ester, epoxy, and phenolic resins, as well as any fiber, can be employed. For resin control, nips and dies are utilized in this process. The winding of filament is a rapid and cost-effective operation. Structure load bearing can be accomplished by the use of complex fiber arrangements. Low-viscosity resin is preferred in this method, as it is in the hand layup technique. Only convex-shaped components may be manufactured using this method. It is difficult to lay fiber along the length of a component. It is used for pipelines, pressure bottles, chemical storage, fire-fighters, rocket engine packing, gas cylinders, and so on [84–88].

3.4. Pultrusion

In this method, composites are recovered as fibers and textiles from a liquid resin solution. A warmed die is used to draw fiber-wetted resins. Finally, the material is cured to its final size and shape. The die is not a duplicate of the last needed item. Then, the finished piece is chopped along its length. Textures can also be shown directly in the die. Fibers are utilized in conjunction with resins, for example, vinyl ester, epoxy, and polyester. This technique is ideal for large-scale manufacturing. It is also a speedy and cost-effective technique. Resins are precisely regulated, and the surface finishing is improved. However, it is limited to a specific sort of cross-section component. The cost of a heated die is significant, and smaller cross-section items, such as beams and girders used in bridges and frame construction, are made using extrusion [37,60,88].

3.5. Braiding

Braiding is a pre-programmed production procedure. To get the appropriate shape, the toes are entangled with one other. The mandrel has a final form of product with interlacing. The toes must be impregnated with resin. The object is then recovered under normal atmospheric conditions or in an autoclave. Furthermore, this interlacing may take place over the mandrel that contains the ultimate shape of the product. The toes would want to be impregnated with resin. The item is then restored at ambient temperature or in an autoclave [88].

3.6. Vacuum Bagging

This is an expanded form of a wet lay-up method. Lamination is subjected to pressure to improve consolidation. This may be performed by affixing the plastic firmly to wet-layup laminates. Heavy resins and fibers such as phenolic and epoxy are utilized to apply one-atmosphere pressure to the laminates through the vacuum pump. Vacuum bagging allows for high fiber-content laminates. Because of the pressure, lower voids are attained, and fibers are properly wetted. The vacuum bag increases safety and minimizes the quantity of volatile emissions [37,60].

3.7. Resin Transfer Molding and Centrifugal Casting

The method entails putting the fibers or fabric materials in the desired direction. These are pre-squeezed to form a shape and are held together with the assistance of a binder. After that, a second coordinating mold instrument is fastened on the first one. At that time, a cavity is filled with pressure resin. The name of the process is Vacuum Assisted Resin Injection (VARI). The laminate has now been restored. Both infusion and fixing can take place at either raised or room temperature. This approach is the most cost-effective and works best for complicated forms. However, the tooling for this process is quite costly and confined to tiny components. This process is used to create tube-shaped components such as motor casings, engine covers, and so on [88].

Under pressure, cleaved fibers and resins are transported to cylindrical moldings in the centrifugal casting technique. A mold is constantly turned. A mixture of resin and cleaved fibers accumulates on the molding wall because of centrifugal action. As a result, the mix takes on the completed shape of a component. This approach is particularly cost-effective and works well for hollow cylindrical goods. However, complicated forms cannot be created in this method, and low-viscosity resin is necessary [83–90].

4. Natural Fiber Surface Treatment

Before being utilized to create composite materials, NFs are commonly subjected to surface treatment to improve their properties. Surface modification of fibers can reduce moisture absorption while raising bonding, roughness, and wettability among the fiber and matrix. As a consequence, it is possible to improve the strength characteristics of the created composites [2,76,91,92]. This study will concentrate on NF since the treatment they get enhances the operation of hybrid composites. Figure 6 depicts the division of surface treatment.

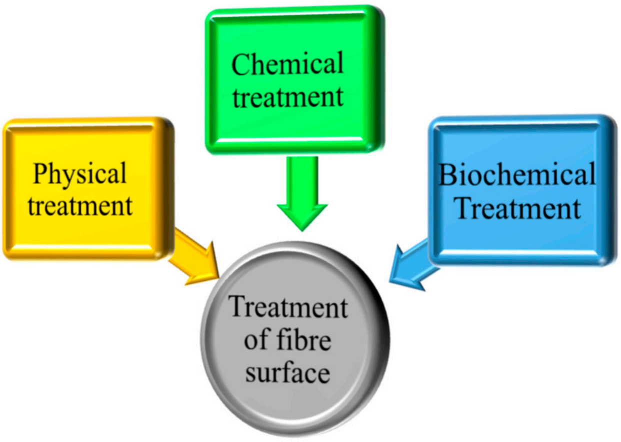


Figure 6. Fiber treatments via various techniques.

The most popular natural fiber surface modification methods are concisely described in this segment (physical and chemical) [2,76,91,92].

4.1. Modifications in Physical Treatment

The most typical physical processes utilized to alter the surface of NF [93] are corona, heat treatments, plasma, fiber, ultraviolet (UV), electron radiation, and pounding. These processes are depicted in Figure 7. The primary requirements for physical processing are the filamentization of the bundles of fibers and exterior alteration of the fibers to increase compatibility through the polymer matrix. By changing the fibers' surface properties while keeping their structural makeup unaltered, these treatments increase fiber–matrix grip [94]. Contrarily, because of the equipment used in exterior alteration processes, physical treatments are more pricy than chemical treatments [95]. Table 3 summarizes the main effects of physical therapy on NF.

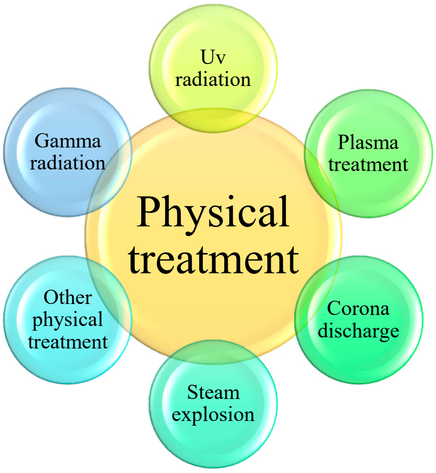


Figure 7. Different types of physical treatment for natural fibers.

The increase in mechanical and thermal characteristics is proportional to the chemical concentration used and the exposure period. In a few cases, combining treatments with two distinct chemicals resulted in superior mechanical and thermal characteristics to the separate treatments. Improved wettability, interface adhesion, and fiber–matrix roughness were seen in NF with physical and chemical surface changes. Furthermore, the majority of treatments seek to reduce natural fiber hydrophilicity and moisture absorption. The ideal fiber modification approach will be determined by the composite end-use application and the fiber matrix employed. Understanding fiber–matrix interfacial characteristics and bonding processes is critical for optimizing NFRC and hybrid composite applications [95]. It will take a lot of investigation to understand this. Figure 7 shows Different types of physical treatment for natural fibers.

4.2. Chemical Treatments

Cellulose, hemicelluloses, and lignin make up the majority of these lignocellulosic fibers. The major element that gives the fiber its strength is cellulose. Many different chemicals are used to treat NF. The primary goal is to improve the fiber cellulose content while eliminating unwanted contaminants, for example, oil and wax [96,97]. Chemical treatments are a typical method for improving fiber–matrix interface adhesion by chemical attachment or minimizing fiber water immersion and mechanically interconnecting at the boundary [96,97]. According to the literature, there are many fiber-changing methods with different levels of effectiveness. Chemical methods frequently outperform physical methods in terms of improving properties. This section covers the details of chemical treatments. The nature of the hydrophobic matrix and the fiber's hydrophilic character produce the bulk of difficulties with natural fiber composites. Due to this inherent mismatch between the matrix and the fiber, mechanical performance is constrained by weak interfacial bonding, and long-term properties are impacted by low moisture resistance [96,97].

An essential factor in achieving enhanced fiber-reinforcement composite properties is the connection strength between the polymer matrix and the fiber in the composite. The fiber absorbs a remarkably high quantity of moisture because of polar clusters and dangling hydroxyl in the fiber, which lead to poor interfacial contact between the hydrophobic matrix polymers and the fiber [98,99]. To create composites in addition to adequate strength characteristics, fibers were chemically treated to minimize their hydrophilic behavior and moisture absorption [98,99]. The goal of these operations is to reduce the number of voids and amorphous areas in the fibers. Because of the enhanced packing of cellulose chains during lignin breakdown, chemically processed fiber-reinforced composites offer better strength properties over untreated fiber-reinforced composites. Figure 8 shows the Different types of chemical treatments for natural fiber.

4.2.1. Acetylation Treatment

Chemicals containing a group of acetyls (CH_3CO), such as acetic anhydride and acetic acid, are added to produce acetylation. The cellulose fibers and the acetyl group interact, resulting in the loss of hydroxyl groups and a decreased capacity to absorb moisture. Acetylation produces a rugged surface topography with fewer voids than other chemical processes, which enhances interlocking properties [100]. Acetylation can be performed with or without the assistance of an acid catalyst, according to Equations (1) and (2), with the inclusion of acetic anhydride resulting in a quicker reaction after the fibers have been soaked in acetic acid. Devoid of the usage of a co-solvent or a catalyst, the lignocellulosic material should be acetylated at 120–160 °C using a tiny amount of liquid acetic anhydride.

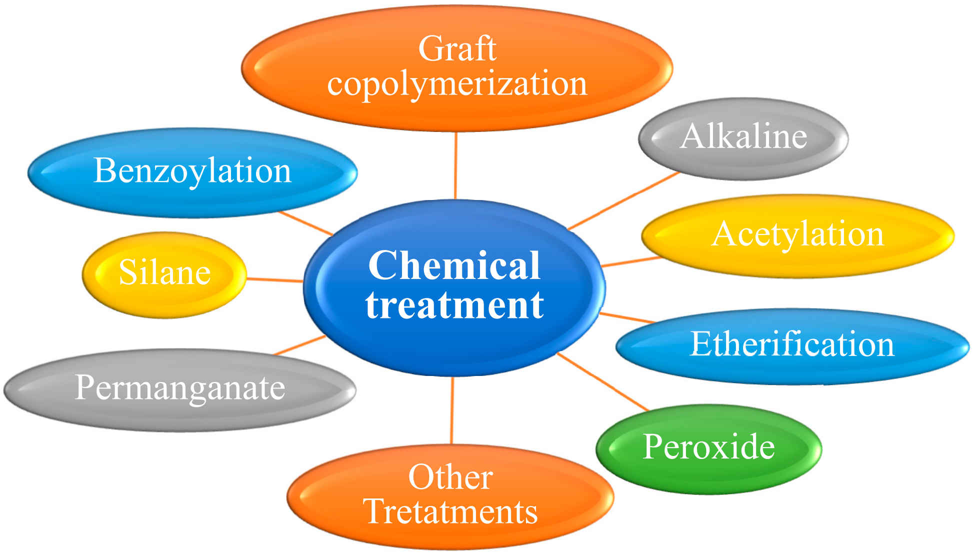
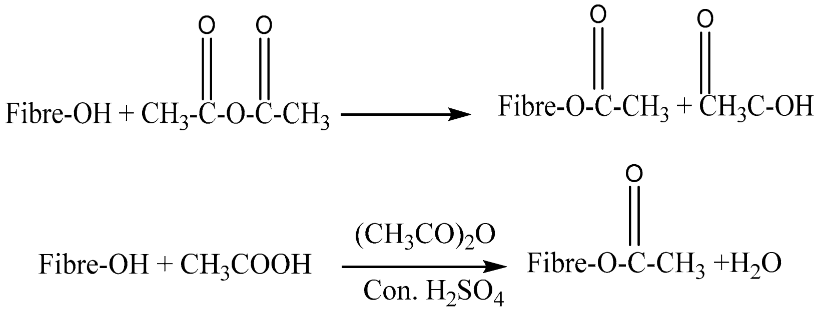


Figure 8. Different types of chemical treatments for natural fiber.

Potentially, small amounts of acetic acid might be used to open the cell wall and initiate the process. According to a tensile strength test, fibrillation happens when binding components are removed from the treated fibers and specific micropores start to appear [101]. Additionally, when the level of acetylation rises, the fibrillation rises as well, leading to splits and deterioration of the fiber. Acetylated fiber composites also have improved resistance to UV radiation degradation, dimensional stability, and resistance to biological assault [102]. Acetylation promotes dimensional stability by reducing water absorption [103]. Additionally, acetylation has been demonstrated to increase the bond shear strength, materials' stiffness, and tensile strength [101,104].



$$\text{Fibre-OH} + \text{CH}_3\text{-}\overset{\overset{\text{O}}{\|}}{\text{C}}\text{-O-}\overset{\overset{\text{O}}{\|}}{\text{C}}\text{-CH}_3 \longrightarrow \text{Fibre-O-}\overset{\overset{\text{O}}{\|}}{\text{C}}\text{-CH}_3 + \text{CH}_3\overset{\overset{\text{O}}{\|}}{\text{C}}\text{-OH}$$

$$\text{Fibre-OH} + \text{CH}_3\text{COOH} \xrightarrow[\text{Con. H}_2\text{SO}_4]{(\text{CH}_3\text{CO})_2\text{O}} \text{Fibre-O-}\overset{\overset{\text{O}}{\|}}{\text{C}}\text{-CH}_3 + \text{H}_2\text{O} \tag{1}$$

$$\begin{aligned} &\text{Fibre} - \text{OH} + \text{KMnO}_4 \longrightarrow \text{Fibre} - \text{O} - \text{H} - \text{O} - (\text{MnO}_2) - \text{O}^-\text{K}^+ \\ &\text{Celluose} - \text{H} + \text{Mn(III)} \longrightarrow \text{Celluose} - \text{H Mn(III)complex} \\ &\text{Celluose} - \text{H} - \text{Mn(III)} \longrightarrow \text{Celluose} + \text{H} + \text{Mn(III)} \end{aligned} \tag{2}$$

Equation (1) Acetylation along and Equation (2) devoid of acid catalyst [101,104].

4.2.2. Acrylation Treatment

Acrylic acid (AC) (CH_2=CHCOOH) is employed to increase polypropylene fiber adherence to their matrix. Hydroxyl radicals on cellulose interact with AC molecules to produce cellulose radicals for the polymerization medium. Peroxide radicals initiate the implanting of AC onto the matrix. Hydrogen atoms are from the hydroxyl groups eliminated from peroxide's O-O bond in cellulose. As a result, as illustrated in Equation (3), these acids produce ester bonds with the hydroxyl groups (2). This fiber–matrix coupling procedure enhances the capacity for stress transmission at the interface and results in improving the composite properties. AC decreases fiber's hydrophilicity [105,106]. At reflux temperature, aqueous 10% AC reacted with alkali-treated fibers resulting in a boost in flexural and tensile strengths by 13.9 and 42.2% in the jute–epoxy-phenolic resin composite [107]. At 24 °C room temperature, the fibers were steeped in 10% NaOH for an hour. The wet product was then subjected to several concentrations of acrylic acid treatment. Additionally, the reaction was run for an hour at 50 °C, which had the effect of lowering the tensile modulus. On the other hand, high extensibility and increased impact resistance were noted [108]. Compared to earlier processes, 40 weight percent fiber at 50 degrees Celsius produced a considerable quantity of moisture absorption capability (50%) [108,109]. In a NaOH solution, flax fibers were soaked for 30 min before spending two hours at 50 °C in an acrylic acid solution. This produced a fiber surface that was very smooth. It has a stronger tensile strength and less water absorption when compared to silane, permanganate, and sodium chloride [110].

$$\text{Fiber} - \text{OH} + \text{C}_2\text{H}_3\text{COOH} \longrightarrow \text{Fibre} - \text{OOCC}_2\text{H}_3 + \text{H}_2\text{O} \tag{3}$$

Equation (3). Acrylation treatment of the fiber [108,109].

4.2.3. Grafting Using Acrylonitrile

Free radicals produced by acryliconitrile (AN) dehydrate and oxidize the cellulose molecules in the fiber. Activated free-radical sites interact with the monomer of the matrix on the fiber surface. Either the available radicals can be produced in reaction media via a redox process and be carried there or the backbone can be directly oxidized by transition metal ions (such as Ce^{4+}, Cr^{6+}, and V^{5+}) [111]. The Equation depicts the entire response (4)

$$\text{Fibre} - \text{OH} + \text{CH}_2 = \text{CHCN} \longrightarrow \text{Fibre} - \text{OCH}_2\text{CH}_2\text{CN} \tag{4}$$

Equation (4). Acrylonitrile grafting treatment of the fiber [111].

The amount of lignin in the fiber has a large impact on the degree of grafting. Lignin, in general, slows polymerization and functions as an inhibitor at greater lignin levels. Grafting can increase several properties, for example, moisture absorption, solubility, chemical and heat resistance, and swelling behavior in various solvents [2,111]. The combination of wet fibers of Agave americana and nitric acid (0.277 mol L^{-1}) containing ceric ammonium nitrate was agitated at the ideal temperature and time interval after the addition of monomer components. The grafted fibers were thoroughly cleaned with distilled water after the homopolymer was removed using dimethylformamide, and they were then dried at 60 °C to get the findings shown below. As the graft percentage grew, so did the resistance to moisture, acid, and base. Graft copolymerization significantly boosted the thermal stability of the fibers [111].

Oil palm-phenol formaldehyde fibers that had been alkali bleached (2 percent for 30 min) and oxidized (0.02 mL^{-1} $KMnO_4$ for 10 min) had also been rinsed in water before being combined in a 30:1 ratio with 1 percent H_2SO_4 containing acrylonitrile. Additionally, it spent 120 min at 50 °C in a thermostatic water bath, where the results showed stronger elastic nature due to higher strain values and a small improvement in stiffness 28 [2]. In comparison to previous treatments, fiber at 40 weight percent at 50 °C generated a greater moisture absorption capacity (200%) [111]. For 10 min, a known amount of butyl acetate, potassium permanganate, concentrated HNO_3, glacial acetic acid, and 0.1 g of pineapple leaf were combined in a container. Before the examination of fibers, it is extracted and completely immersed in N-N dimethylformamide. The findings demonstrate that modified fibers have a poorer graft yield than untreated fibers [2].

4.2.4. Alkaline Treatment

The application of a known concentration of sodium hydroxide (NaOH) onto the NF is known as alkaline treatment (mercerization). Sodium hydroxide interacts with alkali-sensitive hydroxyl groups (OH) on the exterior of fibers, resulting in the release of water particles. The moisture absorption propensity decreases as the number of hydroxyl groups decreases. It also dissolves hemicellulose, pectin, lignin, and another waxy layer in small concentrations. The chemical route between fiber and alkali is depicted in Equation (5), and Figure 9 displays the influence of alkali treatment on NF [112].

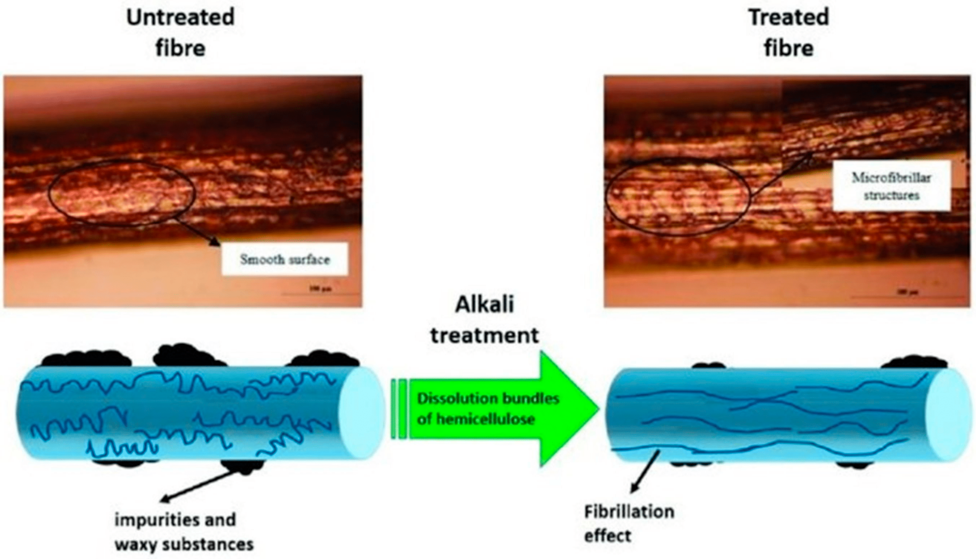


Figure 9. Alkali treatment on natural fiber at 100 µm [112].

$$\text{Fiber} - \text{OH} + \text{NaOH} \longrightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}_2\text{O} \tag{5}$$

Equation (5). Alkali treatment on natural fiber.

Alkali treatment boosts mechanical interlocking at the interface by generating a bumpy surface texture, which improves the adherence of the fiber surface characteristics. This is achieved by disabling hydrogen bonding and removing synthetic and natural impurities from the network structure. Additionally, fiber has increased thermodynamic stability [2,102,113,114]. The alkali procedure removes microvoids from the surface, making it more uniform and boosting its ability to transfer stress [2,102,113,114]. When fibers are considered with sodium hydroxide (NaOH), the aspect ratio and fiber diameter both decrease, which improves the fiber–matrix interactions because more surface area is created because it increases effective surface area and is good at removing waxy coatings. For the pretreatment of fibers, mercerization is widely used prior to supplementary substances being applied for strengthening. Treatment with alkali or NaOH greatly improves mechanical, thermal, and water retention properties [2,102,113,114].

The ideal soaking time and concentration of NaOH solution are those that produce the best mechanical properties. Mechanical qualities have been shown to improve with increasing concentration. Any concentration beyond the recommended level caused harm to the fibers by enlightening them. For improved crystallinity, wettability, amorphous area reduction, and fiber diameter, Tridax procumbens fiber was treated with 5% NaOH [36,115]. The flexural strength and tensile modulus of the NFRCs are also enhanced by alkaline treatment [116–118].

Table 4 gives a general review of the allowable NaOH concentrations and soaking times that result in the strongest fibers before delignification, which causes the fiber to wear out.

Table 4. Effect of sodium hydroxide on natural fiber.

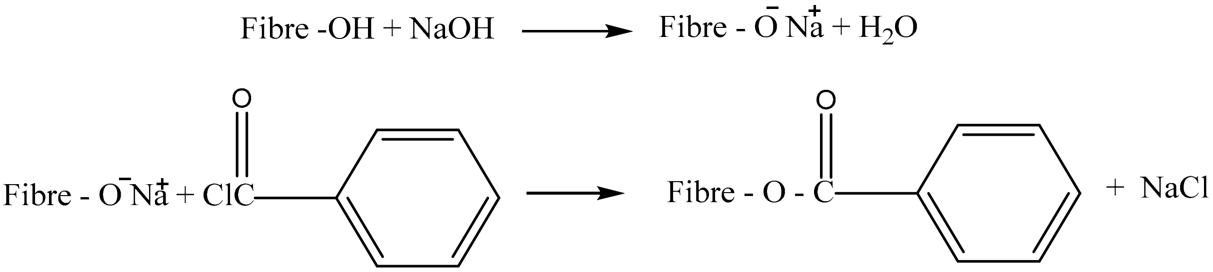
S/n	Experimental Conditions	Matrix–Fiber Used	Outcome	Ref.
1	NaOH (3,5,7 and 10%), 24 h, 70 °C	Epoxy–PALM	Tensile strength, water retention, fiber density, and thermal resistance were improved by ~7% but decreased in fiber diameter.	[119]
2	NaOH 6%, 3 h, 95 °C.	Epoxy–Rice husk/glass F.	48% and 27% increases in flexural and tensile strengths, respectively	[120]
3	NaOH (3,6 and 9%), 3 h, 95 °C	Kenaf Bast	Pure rest surface achieved 9%, but decreases in tensile strength; as such, 6% was the best.	[121]
4	NaOH (3,6, and 9%), 24 h.	MAPP/MAPE	The best result (modulus of elasticity and tensile strength) was achieved at 6%	[122]
5	NaOH 10 %, 1 h.	LDPE–Sisal	Increase in crystallinity and decrease in sorption capacity	[123]
6	NaOH 6%, (45 s, 1 and 45) mins, 21 °C.	PP–Sisal	Shear strength was increased by 173% for the 45 min sample	[124]
7	NaOH (1,3,5%), 2 h,24 °C	Epoxy–Strawberry F.	Best result (thermal stability) was achieved at 3% Solution	[125]
8	NaOH (0,5,10,15,20, and 25%)	PP–Jute	At 10%, tensile strength was increased by 16%	[125]
9	NaOH (5,10, and 15%), 6 h, 70 °C	PLA–Jute	At 10%, tensile strength was increased by 7.7%	[126]
10	NaOH 2%, 2:30 h, 70 °C	Ladies finger F.	Drastic improvement in removal of hydrophilic helicases	[127]
11	NaOH 5%, 2:30 h, 90 °C.	Banana fiber	Young modulus and tensile strength decreases.	[128]
12	NaOH (1,3,5%).	PLA–Banana fiber	Best result was at 3% for increases in impact, tensile modulus, and tensile strength.	[129]
13	NaOH 4%, 2 h.	PF–Banana	Small increases in modulus and tensile strengths and quite large increases (117%) in flexural strength.	[130]
14	NaOH (2 and 10%), 1 h.	PP–Banana	Increases in thermal conductivity at 10% solution.	[131]
15	NaOH 1%, 1 h, 24 °C.	PS–Banana/grass	Tensile strength and flexural properties were increased.	[132]
16	NaOH 6%, 3 h, 95 °C.	Epoxy–Sugarcane fiber	Increase in moisture absorption, increase in flexural and tensile strength	[133]

4.2.5. Benzoylation Treatment

Benzoylation is an efficient technique for increasing the fibers' thermal stability and hydrophobicity. The benzoyl ($C_6H_5C_{14}O$) group, found in the most often used chemical in this treatment, is what causes the treated fiber to be less hydrophilic and interact more favorably with the hydrophobic matrix. Equation (6) indicates that during the operation, an alkali is used to treat the fibers. When extractable fiber components, for example, oils, lignin, and waxes are eliminated, additional reactive hydroxyl groups are visible on the surface [87,134]. These filaments receive BP therapy. The fiber hydroxyl groups are now substituted through benzoyl groups. The surface of the fiber is made rougher by benzoylation, which leads to fibrillation and enhances strength contact along with the polymer matrix. This dramatically increases hydrophobicity and matrix adherence. In a tensile test, advanced fiber led to fiber fracture rather than fiber de-bonding [87,134]. With this method, moisture absorption is reduced while Young's modulus and tensile strength are enhanced. Additionally, it enhances hardness, eliminates voids, and boosts Tg and thermal stability [135–139]. Jute fibers that had undergone alkali treatment were immersed in benzoyl chloride ($C_6H_5CH_2Cl$) solution for 15 min before being soaked in ethanol for an hour before being combined with epoxy, improving the storage modulus and thermal stability.

The flax fibers remained dipped in NaOH solution (10%) for fifteen minutes and mixed with ($C_6H_5CH_2Cl$) before immersing in ethanol for 60 min; the results show LDPE had the best tensile and impact strength, while HDPE had the highest impact severities. The alkali-treated sisal fibers remained drenched in BP and acetone for 30 min, yielding tensile strength saturation at 6% BP.

When compared to silane and peroxide treatments, it resulted in reduced water absorption [93,140]. The fiber surface was found to be smooth. After steeping in a 2% NaOH solution for 30 min, chopped banana NFs (6 mm) were vigorously stirred with benzoyl chloride for a further 30 min. Tensile strength and modulus were both enhanced significantly by 13% and 5%, respectively, while thermal conductivity increased noticeably. However, it is still less than that of fibers treated with alkali and silane [93,140].



(6)

Equation (6). Benzoylation treatment of natural fiber.

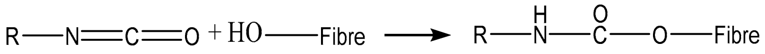
4.2.6. Etherification

By grafting bifunctional monomers, etherification is a chemical process that makes it simple for fiber to react with the matrix polymer chain. Regarding fibers etherified, the thermal steadiness of alfa fiber-reinforced polypropylene composites was considerably improved. Cellulosic fibers are enhanced by being ethylated, which increases their usefulness and acceptance in a number of applications [83]. Sodium hydroxide enhances the benzyl chloride, acrylonitrile, alkyl halides, formaldehyde, and nucleophilic addition of epoxides by producing an alleged medium species including the fiber. Epoxides, like epichlorohydrin, have a stretched, three- or four-membered oxygen-rich ring that removes electrons from surrounding carbons. Because of their structure, epoxides are extremely reactive with substances containing alcohol, such as cellulose [141]. The interaction of epichlorohydrin with wood preservatives such as pentachlorophenol, according to Rowell and Chen, results in wood alteration [141]. Ohkoshi [142] merged dual surfaces of wood that had been altered by allyl bromide etherification by hot pressing.

The shear strength of the preserved wood has been found to be comparable to that of unaffected wood. Furthermore, the effect of allylation on specific wood constituents [143] and the exterior alkylated wood grafted with styrene [142] were investigated. A wood byproduct that could be pushed into films or squeezed out into objects that could be molded was created when benzyl chloride was used in thermoplastic wood. Above 25% of NaOH concentration and temperatures over 90 °C, it was crucial to prepare the wood in order to lessen hydrolysis of the wood's constituents. Wood and bagasse thermo-plasticization was obtained at lower reaction temperatures and with lower alkali pretreatment concentrations. Due to a lower degree of polymerization, wood pulp was shown to be more reactive in a subsequent cyanoethylation study with both cotton and wood pulp [144,145].

4.2.7. Treatment with Isocyanates

Isocyanates are organic mixtures with the N-R=O=C isocyanate functional group. These groups react strongly with hydroxyl groups, resulting in the production of urethane linkages, as illustrated in Equation (7). These are employed in the elimination of hydroxyl sorts through lignin and cellulose. Additionally, the isocyanate interacts with the hydroxyl groups in cellulose to produce urea by combining with the moisture on the fibers. The main disadvantage of alkali treatment is that it causes cellulose hydroxyl groups to lose their hydrogen bonds, making them more reactive than earlier [123]. Isocyanate treatment is seen to be a suitable option for alkalization due to its superior effectiveness at eliminating free hydroxyl groups from the fiber's surface. As a consequence, isocyanate treatment improves strength properties while lowering water absorption [146].



$$R-N=C=O + HO-\text{Fibre} \longrightarrow R-\overset{H}{N}-\overset{O}{C}-O-\text{Fibre} \quad (7)$$

Equation (7). Isocyanate treatment of natural fiber.

Before being treated with isocyanate using dibutyltin dilaurate as an impetus at 125 °C for 1 h in a composite made of flax-epoxy, jute, and hemp, the nonwoven fiber mat was permitted to develop in DMF for thirty minutes. The carpets were extensively washed in hot DMF after the process and dried in a 105 °C oven, yielding a product with enhanced firmness and a 17 percent reduction in influence strength [147]. The results show that at 40 weight percent, fiber at 50 °C had a better dampness assimilation limit (280%) than other medications. The filaments were treated with a soluble base, then immersed in chloroform including toluene diisocyanate. Dibutyltin dilaurate was applied stepwise, and the mixture was unsettled for two hours and cleaned by refluxing with oil palm-pineapple fiber [109]. The alkali-treated fibers were then treated with CCl_4 and a little quantity of dibutyltin dilaurate catalyst. The urethane derivative was poured into the flask stepwise, with steady swirling, until it was completely dissolved. The reaction was permitted to remain for 1 h, and the findings show that the dielectric constant decreased after the treatment [148]. Fibers outperformed alkaline and untreated fibers in conditions of tensile strength, and when matched to alkaline-treated fibers, the elongation at break was doubled [123]. Pre-dried fibers were treated with DIC in another study undertaken by Wulin et al. [149]; prior to the fibrous cellulose–PP composite, the finished product had 30 wt% fibers. The reason for the physical and chemical treatment of natural fibers are shown in Table 5.

Table 5. Reason for physical and chemical treatment of natural fibers.

Treatment Type		Treatment Effect	Ref.
Physical	Corona	Corona treatment alters the fiber surface by oxidizing it and combining polar groups, resulting in cleaner fiber surfaces and improved interfacial grip between the fiber and matrix.	[150,151]
	Gamma ray (γ)	Gama radiation with extremely high energies can alter the characteristics of polymer surfaces. The optimal gamma radiation exposure increases the tensile characteristics of composite materials to a certain amount for usage in various practical applications.	[152]
	Plasma	Plasma treatment, which uses ionizing gases, improves the elimination of pollutants from the fiber surface. This treatment also decreases surface irregularity by improving the interfacial grip of the fiber–matrix.	[93,153]
	Thermal	Thermal treatment heats the fiber ranging from 100 to 200 °C, altering its chemical and physical characteristics, low-temperature constituents, water proportion, and cellulose crystallinity.	[97,140]
	Ultrasound	The ultrasonic approach cleans natural fiber surfaces in two directions: micro-streaming and micro-jetting.	[94]
	Ultraviolet (UV)	The polarity of the fiber is changed by UV treatment, which increases adhesion between the matrix and the fiber and wettability. Furthermore, numerous bonds (C-O, C-H, C-Si, C-F, and C-C) are broken, which aids in fiber cleaning.	[97,140]
Chemical	Acetylation	Acetylation is a treatment that employs acid catalysts (acetic acid and acetic anhydride) to increase interfacial bonding in natural fibers.	[94,140,154]
	Alkaline	Alkalization or mercerization treatment enhances impurity removal and reduces hydrophilicity and fiber components. The treatment enhances its surface roughness, boosts the fiber–matrix interfacial adhesion, and lowers the diameter of the fiber.	[153,155,156]
	Benzoylation	The hydrophilicity of natural fiber is decreased by benzoylation. Benzoyl chloride is employed to increase fiber–matrix interfacial grip and thermal stability. The hydroxyl groups in the fiber are activated by pre-treatment with alkali.	[94,154,157]

Table 5. *Cont.*

	Treatment Type	Treatment Effect	Ref.
Chemical	Etherification	Etherification is a biochemical process that permits fiber to easily alter with the matrix's polymer chain by grafting bifunctional monomers. The thermal stability of alfa fiber-reinforced polypropylene composites was greatly increased when etherified fibers were used.	[6,48]
	Isocyanate	The procedure is utilized in natural fibers as a coupling agent, allowing them to interact with the fiber's hydroxyl groups, enhancing water resistance and interfacial grip.	[97,153]
	Permanganate Treatment	This approach employs the chemical potassium permanganate. The potassium permanganate's MnO_4 group interacts with the natural fiber's cellulose group to generate a complex ion. Graft polymerization is induced by the highly reactive Mn ion. The $KMnO_4$ treatment improved the fibrillation of polythene fibers and roughness, resulting in considerably improved rubber composite characteristics.	[158]
	Silane	Silane treatment results in the formation of a micropore coating of a natural fiber's surface. Improves interfacial grip of the fiber matrix. Pre-treatments, primarily alkalization, are carried out to purify existing components.	[97,159,160]
	Maleated coupling agents (Graft co-polymerization)	This treatment involves the addition of a coupling agent to the matrix and the fiber's surface. To improve the coupling agent and the compatibility of the matrix, maleic anhydride is grafted onto the polymers.	[94,161]

4.2.8. Permanganate Treatment

In acetone, potassium permanganate ($KMnO_4$) produces extremely reactive MnO_4-ions; these ions form cellulose-manganate when they relate with the hydroxyl groups in cellulose. This triggers the graft copolymerization process, which results in the production of fiber with outstanding thermal stability. Generally, soaking (for 1–3 min) is undertaken after an alkaline pretreatment by applying various concentrations of $KMnO_4$ solution in acetone. In addition to removing the hydroxyl groups from the cellulose, it also interacts with the lignin in the cell wall and eliminates it. There is thus a high level of hydrophobicity. Fiber's hydrophilic propensity reduces when $KMnO_4$ concentrations grow to an optimal amount [89,148]. Polar groups between fiber and matrix are produced at a concentration of 1%, resulting in cellulosic fiber breakdown [89,148]. The oxidation of permanganate etches the fiber surface, making it physically rougher; in this situation, the induced mechanical interlocking improves interfacial adhesion while simultaneously improving the contact space between the matrix and fiber [123].

The strength of this fusing of a scratched matrix with the fibers induced by oxidation is comparable to chemical bonding observed with silane treatment [134]. When NFRCs are permanganate-treated, their ductility, flexural strength, impact strength, thermal stability, and flexural modulus are all enhanced [162–164]. $KMnO_4$ improves fiber roughness and converts cellulose hydroxyl groups to carboxyl and aldehyde groups [162]. A sisal fiber was treated with alkali and soaked in acetone for 1 min in permanganate solutions (0.033 percent, 0.0625 percent, and 0.125 percent concentrations). The hydrophilicity of fiber reduced as the quantity of $KMnO_4$ increased. However, the maximum cellulose degradation was seen at a $KMnO_4$ concentration of 1% [148]. In another study, ref. [36], alkali-pretreated fibers were immersed in permanganate solution (conc. of 0.01%, 0.05%, and 0.1%) in acetone for about 2–3 min of the oil palm–PF composite, and in comparison to previous chemical treatments, the findings demonstrate the highest tensile strength and modulus.

A highly fibrillated structure results in an excellent fiber matrix. After 30 min of soaking in a 0.5 percent $KMnO_4$-acetone solution, the alkali-treated banana NF yielded a 16 percent yield. Thermal diffusivity rises [131]. Modulus and tensile strength increased by 7.5% and 6.4%, respectively. Modulus and flexural strength increased by 10% and 5%, respectively, which is less than in alkali and silane treatments [165]. The alkali-treated sisal fibers were immersed in $KMnO_4$ solutions of various dilutions in acetone for 60 s, and the results were quite successful. The cellulose decomposed quicker at 1% concentration, producing more arctic groups compared to unprocessed composites [148]. Sisal fibers were bathed in a 0.055% permanganate acetone solution for 2 min, which resulted in greater inter-laminar shear strength, tensile characteristics, and flexural characteristics than silane-treated fibers; nonetheless, untreated fibers absorb less impact energy [134].

4.2.9. Maleated Coupling Agents

The inclusion of maleated coupling agents improves the interfacial interaction between the matrix and fiber. MA interacts with the hydroxyl groups during grafting and eliminates them from the fiber cells [94]. Figure 10 illustrates how this leads to a long-chain polymer that is covalently bound to the fiber surface. Before the treatment, the copolymer is warmed to 170 °C and then the esterification operation is carried out. Following this treatment, the cellulose fiber's surface energy is significantly closer to that of the matrix. Enhanced wettability of the fiber causes an increase in interfacial adhesion [36]. A separate research study looked at the utility of MAPP in improving thermal deflection temperature, flexural strength, tensile strength, and impact energy absorption when mixed with short Latania fiber at an ideal concentration of 2 wt% [166]. Huang et al. proved the efficiency of wood–polypropylene composites in increasing flexural strength and decreasing water absorption in their paper [167]. Maleated polyolefins (MaPOs) span the gap between polar and nonpolar species thanks to a unique confluence of properties. This, together with the fact that they are easy to make, contributes to MaPOs' popularity [168].

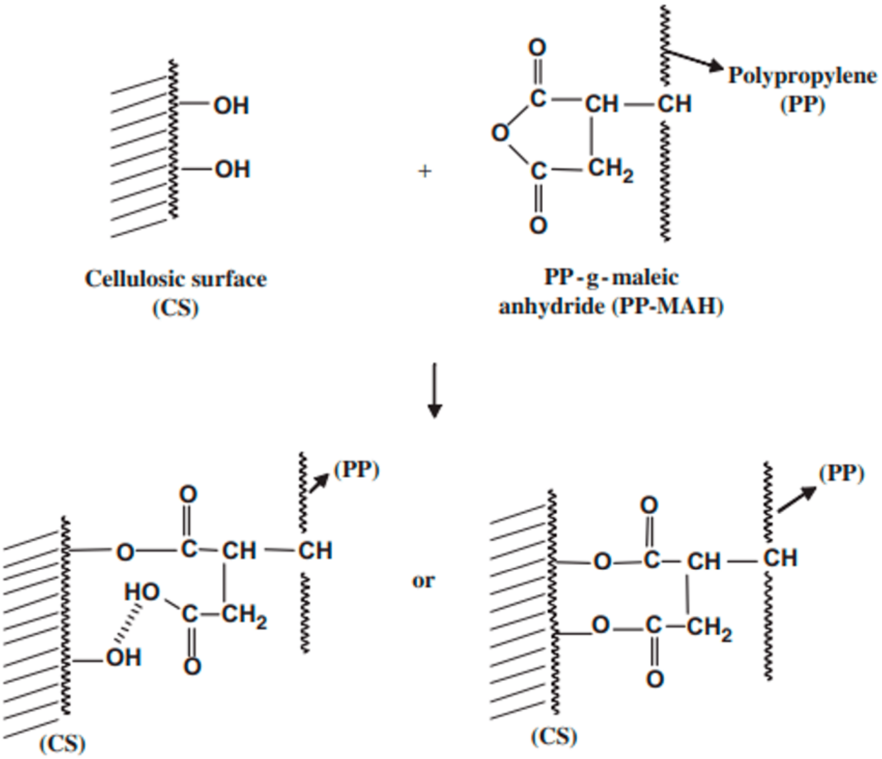


Figure 10. Illustration of coupling agent polypropylene-g-anhydride maleic acid (PP-MAH) and potential esterification process and H-bond interactions at the interface of a cellulosic fiber [169].

4.2.10. Treatment with Peroxide

The divalent O-O ion is a component of functional groups called organic peroxides (RO-OR). These peroxides are highly susceptible to oxidizing into free radicals. As seen in Equation (8), these extremely reactive available radicals react with hydrogen in the hydroxyl groups.

$$\begin{gathered}\text{OR} + \text{RO} \longrightarrow 2[\text{RO}] \\ \text{Fiber} + \text{RO-H} \longrightarrow \text{Fiber} + \text{R-OH}\end{gathered} \tag{8}$$

Furthermore, when the free radical combines with a matrix molecule, such as polyethylene (PE-H), the reaction described in Equation (9) occurs, yielding polyethylene (PE).

$$\text{PE} + \text{RO} — \text{H} \longrightarrow \text{PE} + \text{ROH} \tag{9}$$

Equation illustrates how the PE radicals may interact and cross-link or graft onto cellulose fibers (10)

$$\begin{aligned}&\text{PE} + \text{PE} \longrightarrow \text{PE PE}\\&\text{Fiber} + \text{PE} \longrightarrow \text{PE-Fiber}\end{aligned} \tag{10}$$

Dicumyl peroxide ($(C_6H_3C(CH_3)_2O)_2$) and benzoyl peroxide ($(C_6H_5CO)_2O_2$) are two organic peroxide treatments which are often applied. When composites are being cured, the peroxide breakdown proces takes place at the contact. Higher temperatures encourage the decomposition of peroxides [108]. For a certain fiber composition, the composite's tensile strength obtained its highest value at a critical peroxide concentration. At this stage, grafted polymer (matrix) molecules have been applied to the fibers, and further peroxide addition increases the cross-linking of the matrix molecules, but this does not have influence on the mechanical characteristics of the composite [123]. Water absorption is reduced while thermal stability, interfacial adhesion, tensile strength, and elastic modulus between the fiber and matrix are enhanced by peroxide treatment.

An organic peroxide treatment that is often utilized is dicumyl peroxide ($(C_6H_3C(CH_3)_2O)_2$) and benzoyl peroxide (BP, $(C_6H_5CO)_2O_2$). The peroxide disintegration process happens at the contact during the drying of composites. The breakdown of peroxides is promoted at higher temperatures [108]. For a particular fiber composition, there is a crucial peroxide dilution through which the composite tensile strength attains the maximum. In this phase, the fibers have been coated with grafted polymer (matrix) molecules, and consequent peroxide supplement adds to certain matrix cross-linking, which has no influence on composite strength properties [123]. Peroxide treatment thereby decreases water absorption while boosting interfacial adhesion between the matrix and fiber, thermal stability, tensile strength, and modulus of elasticity [85–87,110,170,171].

4.2.11. Silane Treatment

As a coupling agent, silane (SiH_4), a versatile chemical, is utilized to increase the fiber–matrix interface. Several phases of bond formation, condensation, and hydrolysis occur throughout the fiber treatment process. Silanol is produced in the presence of moisture. Equation (11) depicts the reaction process.

$$\text{CH}_2\text{CHSi}(\text{OC}_2\text{H}_5)_3 + \text{H}_2\text{OCH}_2\text{CHSI}(\text{OH})_3 + 3\text{C2H}_5\text{OH} \xrightarrow{\text{Condensation}} \text{CH}_2\text{CHSI}(\text{OH})_3 \tag{11}$$

When silanol condenses, one side of the substance interacts with the functional group of the matrix, while the other side alters the cellulose hydroxyl group of the fiber to establish a chemical connection (12).

$$\text{CH}_2\text{CHSI}(\text{OH})_3 + \text{Fiber} — \text{OH} \longrightarrow \text{CH}_2\text{CHSI}(\text{OH})_2\text{O} - \text{Fiber} + \text{H}_2\text{O} \tag{12}$$

In general, silane treatment begins with a silane derivative (usually amine) in acetone/alcohol/solution. When the fibers are immersed in the silane solution, they interact with the matrix more effectively compared to alkaline-treated fibers, resulting in higher thermal stability, flexural stiffness, tensile strength, and tensile modulus. Sisal fibers were treated for 2 h with a 2 wt% solution (pH 3.5, room temperature), resulting in an epoxy composite with better impact strength than alkalized fibers. Valadez et al. [172] and Agrawal et al. [173]. Additionally, silane solutions at concentrations of 0.033 and 1% were used to treat palm oil and henequen fibers, respectively. Crystallinity proportion and interfacial interactions were shown to be improved. Fibers were treated for 1 h with a 1 percent oligomeric siloxane 96 percent alcohol solution. Comparing alkali treatment alone to alkali treatment with silane treatment, modulus and strength improved by 7% and 12% [174]. In another research study, the author used natural hemp and basalt fibers that had been treated with cyanate ester and benzoxazine, respectively. According to the researchers, flexural strength, micro-hardness, and thermal stability all increased [175,176]. Atiqah and co. studied sugar palm fibers treated with silane, alkaline, and a silane–alkaline combination. Improved fiber–matrix bonding was achieved by eliminating the outer layers, which include contaminants, fewer nodes, wax, and pectin [177].

4.3. Biological (Fungi and Enzymes)

The above-mentioned chemical treatments for surface modification have several advantages, but they are hazardous and might cause difficulties for the environment. Enzyme therapy is a modern biochemical treatment procedure that is gaining popularity due to its environmental friendliness. Enzymes primarily target non-cellulosic components of fiber; also, enzymes may be regenerated and are cost-effective [2,32].

5. Applications

In recent years, an increasing number of vehicle models, initially in Europe and then in North America, have used natural fiber-built polymers in entryway boards, bundle plates, cap racks, instrument boards, interior motor covers, sun visors, boot liners, and other highly demanded parts, for example, seat backs and exterior underfloor framing. This trend was prompted by government regulation [82].

All of the world's leading automakers now utilize these materials, and their use in this industry is projected to grow [82]. Azman et al. [178] completed a thorough summary of the application of natural fiber composites. Medium-density fiberboard, which has been evaluated for use in railcars, has been replaced by composite board, which was developed in India [39]. NFCs are also being used in the aviation sector for interior panels. They have been used as a synthetic fiber substitute in a number of products, including toys, packaging, nautical railings, and coverings for electrical devices such as laptops and cell phones [179]. Surfing looks to be a standout among sports when it comes to using ecologically friendly materials. NFC-enabled surfboards are currently being advertised by several firms. One of the first was Laminations Ltd.'s "Ecoboard," which used bio-based glue and hemp fiber [39].

According to modern research, RTM may be used to produce natural fiber surfboard fins that are both mechanically and economically viable. CelluComp Ltd., which extracts nano-cellulose from root vegetables, has also created material for fishing poles [39,41]. Sandwich structures have been utilized to make a variety of industrial materials. They are notable for providing increased stiffness-to-weight and strength ratios and are good at absorbing energy and lightweight, allowing for the elimination of some core constituent components through design integration [180]. The Asia 2013 Innovation Award was given to the world's first operational flax composite wind turbine blade by the JEC Composites Group [181]. Recent studies have shown that glass fiber-reinforced RTM flax-reinforced polyester turbine blades can be replaced. Research has also shown that NFCs may be useful for string musical instrument top-plates [182]. Fiber/PE or wood fiber/PP has been widely utilized in decking in the building business, particularly in the United States. NF-reinforced compounds are also employed in non-structural building products such as wall insulation, floor lamination, window frames, and doors [183–186]. NFCs were discovered to have better mechanical qualities than timber laminates in insulating structural panels when the flexural performance of squeeze out cavity cross-section WPCs containing 50 m percent wood floor was evaluated to see if natural fiber composite sheet piles could be used [187,188]. The findings showed that natural fiber light-duty sheet piling constructions have a lot of potential to replace concrete and steel sheet piles. Similar materials have also been suggested for use in beams and slabs [188]. The use of NF to reinforce cement in construction materials is also being investigated [82].

In general, the worldwide NFCs market was valued at US$2.1 billion in 2010 and was anticipated to increase at a rate of 10% per year through 2016, indicating that there is still room for growth in a variety of industries, including civil, construction, sports, automotive, aircraft, and leisure [41,179]. The composites are made utilizing a filament wounding process and a natural fiber core structure. The incorporation of a honeycomb core into a composite tube increases strength and stiffness, which may be advantageous in cross-arm applications [189]. Light airplane radomes must be made of composites with high toughness and a low dielectric constant. According to Haris et al. [190], hybrid deal with glass-reinforced epoxy composites may be a promising substance for radome use on the basis of an initial examination of NF anthology data including kenaf, banana, PALF, bamboo, and palm oil [52]. On military vehicles, a ballistic composite is frequently utilized for the components of shields, body armor, vests, and helmets. Ultra-high-molecular-weight polyethylene (UHMWPE), Twaron, high-molecular-weight polyethylene (HMWPE) (Dyneema), and Kevlar are the most often utilized composite ballistic materials (Spectra) [112,191]. Figure 11 shows the application of various Natural Fibers.

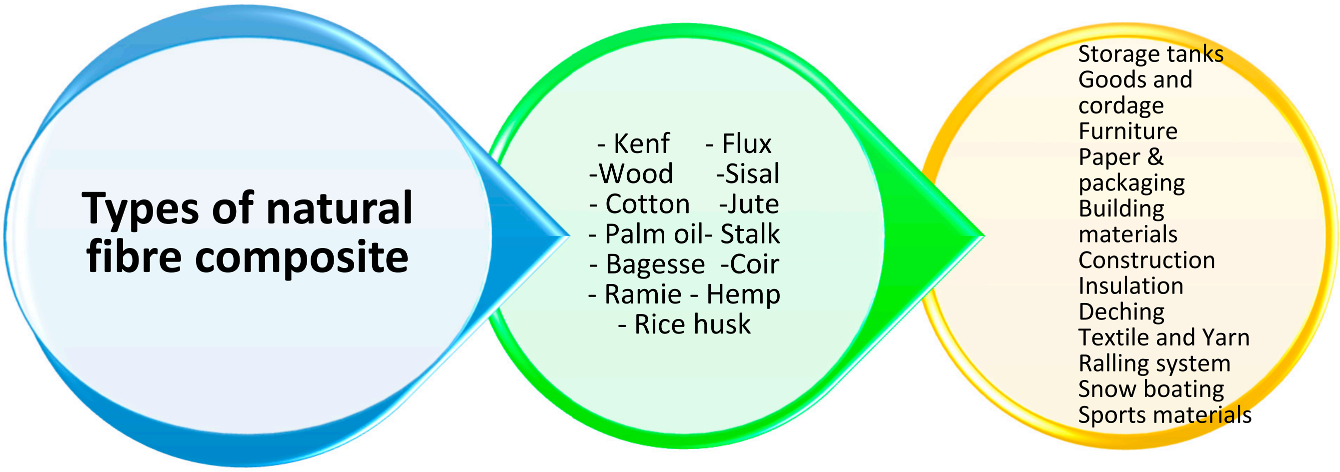


Figure 11. Application of various natural fibers.

6. Conclusions

- NFs, also recognized as plant cellulosic filaments, are increasingly utilized as replacements in composite materials. NFs may represent a significant improvement in the public GDP of agro-based economies since they are easily accessible, affordable, and biodegradable, with a high strength-to-weight ratio and no risk to the population.
- NFRPCs were found to have considerably superior ballistic opposition and energy retention. Engineered strands outperform ordinary filaments in terms of stiffness, thermal properties, and strength. Natural/synthetic hybrid composites show better performance when compared to natural fiber composites.
- Hybridization may allow the use of NFs as composite reinforcement. Their commercial qualities will depend on the development of manufacturing standards and post-treatment techniques.
- Manufacturing standardized fibers may help to avoid the use of NFs with uncertain mechanical and chemical properties.
- From the years 2012 to 2020, a substantial variation in wood-based polymer composites and quantity of natural fiber is observed.
- It may be claimed that without strong government policies to boost the amount of bio-based materials used in high-end products, such as those produced by the automotive, marine, and aerospace industries, their rapid development would not take place.

Future Prospectives

- ➢ NFs can be used as reinforcements made through additive manufacturing. This allows scientists and engineers to construct complex geometric components and functionally rated composites with suitable specific use and operation. These are the advantages of employing additive manufacturing to produce NFRCs and hybrids.
- ➢ NF may be utilized in manufacturing single-use or disposable items to create parts that are individualized, renewable, recyclable, and non-toxic.
- ➢ The benefits of NF versus synthetic fibers are especially notable in terms of printer durability and production costs.
- ➢ Some problems with NFs made through additive manufacturing are the preparation of composite filaments, the prevention of voids from forming, the alignment of the fibers, and how much moisture is involved in the process.
- ➢ These problems can be solved by modifying the hardware, feedstock quality, and additive manufacturing process settings. When combining NF with additive manufacturing, businesses can move towards a wise eco-conscious future through the development of further sustainable material alternatives.

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