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Review article

AGRO-WASTE NATURAL FIBER SAMPLE PREPARATION TECHNIQUES FOR BIO-COMPOSITES DEVELOPMENT: METHODOLOGICAL INSIGHTS

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Abstract. *In today's engineering industries, there is a growing focus on sustainable and eco-friendly products due to their recyclability, abundant availability and property variability. One key aspect of this sustainability effort is the development of eco-friendly materials, particularly bio composites derived from agricultural waste residues. The physical, mechanical and thermal properties as well as suitability of these fibers depend greatly on the methods used for extraction, processing, chemical modification and physical treatments. Understanding these processes comprehensively is essential for obtaining desired natural fibers/fillers from agricultural waste for creating effective bio composites to meet specific application demands. This study aims to provide a comprehensive assessment of the various extraction and modification techniques employed for natural fibers. It offers an in-depth review of diverse extraction processes, ranging from the initial harvesting to the decortication stage. It has been revealed that the choice of extraction methodologies depends on climate, water resources, local traditions, and the desired fiber quality. Additionally, the paper explores chemical and physical treatments, highlighting how each method influences the structure and properties of natural fibers. Overall, this review offers practical insights into the steps taken to transform agro-waste biomass into desired natural fiber and in turn biocomposite material, while enhancing product quality and performance.*

Key words: *Natural fibers, Extraction processes, Chemical treatments, Physical/surface modifications, Bio-composite development*

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1. INTRODUCTION

In response to the escalating global apprehension and consciousness regarding environmental issues, numerous industrial sectors have progressively shifted their focus towards the adoption of sustainable and regenerative materials. Natural fibers, being inherently renewable and readily accessible within the natural ecosystem, have emerged as a pivotal avenue in this pursuit. This inclination is largely attributed to their array of advantages, including but not limited to, cost-effectiveness, lightweight nature, inherent renewability, biodegradability and an array of distinctive high-specific properties [1–3]. With the rise of agriculture, deliberate cultivation of fiber-yielding plants became established. Early civilizations explored plant fibers, particularly in textile weaving, long before perfecting practices like metalworking or pottery. Various types of fibers held significant importance for their versatility. Some were used for crafting ropes and yarns, essential for activities like hunting, fishing, netting, climbing, and transportation, while others found their place in the intricate art of textile weaving [4]. Comprised mainly of lignocellulosic materials, natural fibers encompass a variety of resources, including wood, ramie, jute, hemp, kenaf, sisal, coir, flax, bamboo and various fruit fibers. Effectively integrating natural fibers into composite materials is crucial for their practical application. Substituting synthetic materials with natural fibers aligns with the principles of environmental stewardship, reflecting the contemporary shift towards sustainability. Table 1 presents a comparison of attributes between natural and synthetic fibers. While natural fibers generally exhibit lower tensile strength and modulus compared to synthetic counterparts, they offer significant cost and ecological advantages. Variations in fiber structure and growth conditions contribute to the diverse mechanical properties observed in natural fibers. Through precise treatment procedures, these fibers can be tailored to serve as reinforcing fillers with various mechanical characteristics. Moreover, careful application of specific treatments can even impart mechanical properties similar to those of synthetic fiber composites.

Table 1 Assessment of key properties of major synthetic and natural fibers [5,6].

	Fibers	Density (g/cm^3)	Tensile Strength (MPa)	Elastic Modulus (GPa)	Decomposition Temperature (°C)	Cost (Euros/Kg)
Synthetic	Carbon	1.8-1.9	2090-5200	525	3647	33-166
	Kevlar (Aramid)	1.4	2380-3100	124	496	16.7
	Zirconia	5.6	700	100	2497	41.7
	E-glass	2.5	1625-3400	72	756	1.6-2
	C-glass	2.5	2800	69	756	9.3-16
Natural	Sisal	1.4	507-855	24	270	0.7
	Flax	1.4	250-1000	12-100	280	0.15-0.76
	Cotton	1.5-1.6	350	11	246	0.35
	Banana	1.3	791	30	320	0.7-0.9
	Jute	1.3-1.5	187-540	3-55	270	0.7

The environmental significance of natural fibers lies in their seamless integration with agricultural waste resources, promoting a holistic approach to waste management and valorization. This convergence taps into the untapped potential of discarded agricultural by-products, granting them secondary utility and enhancing the sustainability of resulting composites. The global surge in waste production, fueled by rapid urbanization, population growth, and industrialization, has reached alarming levels. This surge encompasses various waste streams, including agricultural residues. Table 2 provides an overview of the promising agricultural product resources and their respective countries of origin. These residues, once regarded as mere by-products, hold the significant potential to be transformed into valuable natural fibers. Leveraging these residues addresses the dual challenge of waste reduction and resource optimization, offering a pathway to mitigate environmental impact. Agricultural residues fibers encompass a diverse array of chemical substances, predominantly composed of cellulose, hemicellulose, lignin and smaller quantities of pectin [7]. The composition and proportions of these components can vary depending on the type of plant species, climatic conditions and the method of extraction and fiber preparation approaches employed.

Numerous comprehensive studies have evaluated the properties of agricultural waste-based composites [8–10]. These investigations include the isolation and characterization of agro-waste materials, involving analyses of their physical, mechanical and chemical attributes. However, a notable research gap exists in the exploration of agro-residues and their associated extraction and preparation strategies employed prior to their deployment into bio composites. This gap underscores the need for a more comprehensive understanding of the crucial pre-processing steps involved in optimizing the utilization of agricultural residues in biocomposite materials. Achieving optimal performance in bio composites materials requires a comprehensive understanding of the interplay between fiber characteristics and processing techniques. By examining the techniques for fiber extraction, cleaning and modification, along with their impact on agro-waste fibers and their composite performance, this paper seeks to shed light on the intricate relationship between preparation methods and the final properties of the resulting composites. By understanding the fiber preparatory techniques, merits and obstacles, researchers, engineers and practitioners will be better prepared to fully exploit the capabilities of materials founded on natural fibers.

Table 2 Production statistics of agricultural products that can be used as potential natural fiber resources [11]

	Production (million tons)							
Countries	Banana	Pineapple	Coconut	Sugarcane	Rice	Kenaf	Jute	Oil palm
India	24.87	1.46	11.93	341.2	159.2	0.12	1.98	-
China	10.55	1	0.25	125.54	203.29	0.08	0.17	0.67
Philippines	9.23	2.4	15.35	31.87	18.44	-	0.002	0.47
Brazil	6.9	2.48	2.82	0.74	11.76	14.2	26.7	1.34
Indonesia	6.19	1.78	18.3	33.7	71.28	4.35	0.007	120
Thailand	1.65	2.65	1.01	100.1	38.79	1.3	0.06	12.8
Vietnam	1.56	0.54	1.31	20.08	44.04	8.2	0.02	-

2. AGRO-RESIDUES AS SOURCES OF NATURAL FIBERS

On a global scale, roughly 140 gigatons of biomass are discarded as waste annually, contributing significantly to environmental challenges such as the release of greenhouse gases and the deterioration of soil-water quality [12]. As the world's population continues to grow and debates surrounding the allocation of resources for food and fuel intensify, immense pressure is placed on the agricultural sectors to ramp up their agro-food production. This heightened demand and supply of agricultural products have consequently led to a surge in the disposal of biomass residues. Given these circumstances, the recycling and the secondary utilization of this waste have become a critical issue with notable social and economic implications. To address this, the strategic use of agricultural residue waste as natural fiber resources can be approached through three primary avenues: incorporating them as additives to improve compatibility among principal components, reinforcing them within plastic matrices to enhance structural integrity and utilizing them as cost-effective and renewable filler materials. Agricultural residues can be categorized into two distinct groups: field residues, which are remnants left in agricultural fields after crop harvesting (examples include pineapple leaves, cornhusks, cotton seed pods, kapok seed pods and cotton stalks), and process residues, which are materials generated after processing crops into usable resources (examples include bagasse, pea peel, wheat straw and rice straw). Table 3 provides an overview of some major agricultural residues harnessed for their natural fiber attributes [13].

Table 3 Overview of prominent agro-residues utilized for natural fiber resources.

Agro-residues	Synopsis	Fiber source	Utilization
Rice straw	Rice, a staple food for most of the world's population, contributes not only to sustenance but also to a significant agricultural residue known as rice straw. The leftover rice straw presents a valuable opportunity as a source of natural fibers with diverse applications. About 731 million metric tons of rice straw are generated globally each year.	Rice straw comprises the stalks and leaves of rice plants left behind after the grains are harvested. The primary fiber source within rice straw is the vascular bundles located within the stem.	Used for making paper, packaging materials and handicrafts.
Wheat straw	This is a by-product resulting from the collection of wheat grains, consisting of segments between nodes, nodal points, foliage, husks, and central stem parts of wheat. Around 600 million metric tons of wheat straw are generated globally annually.	Wheat straw encompasses the plant material that remains after wheat grains have been collected. Main part is the vascular bundles found in the stem.	Primarily used in paper production and as packaging material.
Corn cob and husks	These maize/corn cultivation residues are typically removed and discarded before consuming the corn. Corn husks and cobs contribute to a substantial portion of agricultural waste, but specific statistics may vary by region.	The corn cob is the innermost part of the corn plant, while the corn husks is the outer layer of green leaves that envelop the corn.	Used for crafts, textiles and cellulose for paper production.

Sugarcane bagasse	Sugarcane bagasse is the aftermath of the sugarcane crushing process to extract the sugary juice. What is left behind is the fibrous material that comprises the stalks' outer layers, mainly consisting of the fibers and pulp that provided structural support to the plant. Global sugarcane production yields about 1.9 billion metric tons of bagasse annually.	Fibrous residue left after sugarcane juice extraction.	Used for paper production, packaging and as a biomass source for energy generation.
Coconut husks	This refers to the coarse and fibrous exterior layer that encases the coconut fruit. This protective covering acts as a natural shield for the inner seed of the coconut. The roughness and texture of the husk are due to the dense network of fibers that make up its composition. Coconut husks contribute to a significant portion of waste from coconut processing, with specific quantities varying based on coconut production levels.	Outer fibrous shell of coconut.	Used for ropes, mats, brushes, geotextiles, and horticulture substrates.
Pineapple leaves	Pineapple leaves, an often-overlooked component of the fruit-bearing plant, hold remarkable value beyond their decorative appearance. These leaves, situated at the crown of the pineapple plant, are a source of natural fibers known as piña fibers. Pineapple cultivation gives rise to a notable quantity of plant waste, about 250 metric tons per hectare.	Leaves of pineapple plants.	Used for delicate textiles like piña cloth, as well as crafts.
Fruits and vegetable waste	Fruits and vegetable waste refers to the discarded or unused parts of fruits and vegetables that are generated during the harvesting, processing, or consumption of these food items. Estimates of global food waste, including fruits and vegetables, have suggested that roughly one-third of all food produced for human consumption is lost or wasted.	Peels, skins, stems and other parts not consumed as food.	Used as growing mediums for plants, helping improve soil structure, water retention, and aeration in horticultural settings.

3. NATURAL FIBER EXTRACTION PROCESSES

Natural fiber extraction serves as the fundamental and pivotal initial phase in the broader realm of fiber processing. This process encompasses a series of techniques aimed at isolating fibers from various plant sources [14]. Achieving this separation requires the delicate task of disrupting the connections or bonds between the robust stem cores and the delicate fiber bundles surrounding them. The broader context of the fiber extraction process extends beyond just straw; it encompasses processes of liberating fibers from diverse plants components. These methods play a crucial role in enhancing the quality, durability and usability of these fibers. In the context of agricultural waste, the residues undergo preparatory steps to make fiber extraction more efficient. Depending on the type of residue, this might involve harvesting, cleaning, drying and removing any unwanted materials like dirt or impurities. Figure 1 provides an overview of the key processes involved in the extraction of natural fibers for the development of bio composites.

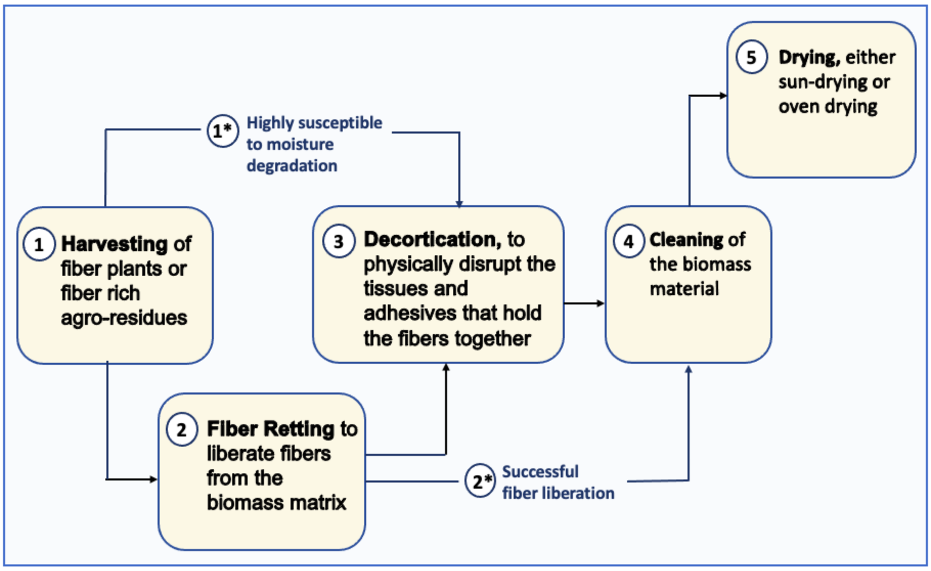

Fig. 1 Processes involved in the extraction of natural fiber from plant biomass to use as reinforcement phase in bio composites.

3.1. Harvesting

The process of harvesting natural fibers from various agricultural crop residues involves intricate systems that are often tailored to local conditions and practices. These systems are designed to effectively extract fibers from plants while considering factors such as the type of crop, the quality of the fibers needed and the prevailing agricultural practices in the region. As a result, harvesting methods can vary significantly depending on these factors. The development of harvesting systems is not a one-size-fits-all approach [15,16]. Rather, it is a dynamic process that considers the unique characteristics of each crop, the resources available and the expertise of local farmers. This localized approach ensures that the methods used are efficient, sustainable and suited to the specific agricultural context. One of the primary distinctions in harvesting methods lies in the choice between manual and mechanized approaches as shown in Figure 2. Manual harvesting is prevalent in many regions, particularly where labour is readily available and cost-effective. Skilled workers harvest fibers by hand, employing traditional techniques that have been refined over generations. In contrast, mechanized harvesting involves the of technology and innovation. This harvesting method have gained popularity in areas where labour costs are higher or where the demand for efficiency is paramount. Specialized machinery is used to streamline the process, reducing labour requirements, and increasing overall output.


a) b)

Fig. 2 Comparison of manual harvesting and machine harvesting techniques in the context of natural fiber extraction from agricultural crops; (a) Manual harvesting, showcasing skilled workers meticulously handpicking fibers from the plants, (b) Machine harvesting, where specialized machinery efficiently gathers fibers from the plants.

Moreover, within harvesting systems for natural fibers, two distinct methods often come into play: whole stem harvesting and cut stem harvesting. These methods are indicative of the intricate decision-making processes involved in optimizing the extraction of fibers from various agricultural produce. The whole stem harvesting approach involves gathering the entire plant, including the stalk, leaves and sometimes even the roots to extract fibers [17]. This method can result in minimal waste, as nearly all parts of the plant are used in some way. It can also simplify the processing steps, especially when the fibers are distributed along the entire length of the stem. In contrast, cut stem harvesting involves severing only the part of the plant that contains the desired fibers, often leaving behind leaves and other non-essential portions [18]. This method is frequently used for crops like sugarcane and bamboo.

3.2. Fiber Retting

Natural retting involves the controlled decomposition of non-fibrous materials that bind the fibers to the plant's stem. Through retting, this plant structure matrix is deliberately broken down, effectively loosening the fibers and making them accessible for further processing. A comparison between retting methods is shown in Table 4 [19–21]. The outcome of this transformative process has dual effect. First, the liberated fiber bundles, now freed from their binding matrix, emerge in a state that allows for their separation with greater ease. This sets the stage for subsequent processing steps, such as scutching and hackling, that refine the fibers and align them in a more uniform manner. Secondly, the residual fibers that remain after retting become enriched in cellulose content, a crucial factor contributing to the high-strength properties of the extracted fibers [22].

Numerous extensive inquiries and studies have been dedicated to comprehending the intricate influence of retting processes on the characteristics of natural fibers and their respective composites. Stawski et al. [23] examined the thermal and mechanical attributes of Okra fibers acquired through water retting and dew retting methods. These fibers were subjected to different processing techniques for the removal of non-fibrous components. Yu et al. [24] conducted a study involving the use of warm water retting as a pre-treatment method for banana stems. The research aimed to assess how varying durations of retting treatment impact both the physical and chemical properties of both the banana stems and the fibers derived from different parts of the plant. The findings revealed that longer retting periods were associated with a reduction in the presence of fiber impurities. However, this prolonged retting time also led to a decline in the strength and toughness of the resulting fibers. In their study, Narkpiban & Poonsawat [25] explored the impacts of hydrothermal pre-treatment on the characteristics of kenaf fibers subjected to water and enzyme retting. This involved treating the fibers in a high-pressure reactor at temperatures of 160, 180, and 200°C. The findings revealed that both water and enzymatic retting led to an increase in cellulose content within the fibers.

The implications of retting processes are not confined solely to the fibers themselves. The interactions between retted fibers and the matrix materials used in bio composites are a matter of considerable interest. For instance, De Prez et al. [26] conducted a study focusing on the mechanical properties of composites that were strengthened using fibers extracted after undergoing enzymatic treatments. The investigation also delved into factors such as fiber fineness, moisture absorption in the composites and the analysis of fracture surfaces. The findings indicated that all the enzymatic treatments exhibited encouraging results in terms of tensile strength and stiffness. Moreover, Rozyanty et al. [27] examined how different retting methods (water and mechanical retting) for kenaf bast fibers impact the tensile strength and water absorption properties of unsaturated polyester (UPE) composites reinforced with these fibers. The study revealed that water-retted kenaf bast fibers possess surfaces characterized by cleanliness, smoothness, minimal fiber detachment and fewer gaps compared to the mechanically extracted samples.

Recent practices have witnessed the integration of advanced technologies and pre-treatment methods into the retting process, exemplified by a study conducted by Ruan et al. [28]. In their research, they employed Radio Frequency (RF) pre-treatment to effectively break down non-cellulosic components present in the material. This pre-treatment process was particularly advantageous as it prompted the rapid absorption of water and expansion of the polar constituents within the flax phloem. As a result, this action effectively loosened the connections between individual fibers and fiber bundles, creating a more accessible pathway for enzymes to penetrate the material. The outcomes of their investigation revealed that the implementation of RF pre-treatment had a positive impact on the overall retting process. The enhanced breakdown of non-cellulosic materials and the improved water absorption and swelling characteristics contributed to an increase in retting efficiency. The choice between the retting methodology depends on factors such as climate, available water resources, local traditions and the desired quality of the fibers. Proper timing and monitoring are crucial in all methods to ensure that the fibers are neither under-retted which can result in insufficient fiber separation nor over-retted leading to fiber damage. Réquilé et al. [29] conducted a study that affirms how the desired fiber quality in hemp can be ensured through the retting process by adjusting the duration of the retting process based on various weather conditions, such as the strength of solar radiation, the amount of rainfall and the surrounding temperatures. In situations where there is a shortage of rainfall or the air temperature is low, it becomes necessary to extend the retting period to allow for the attainment of an adequate level of retted biomass, which is crucial for achieving the desired fiber quality. Retting not only facilitates fiber separation but also has environmental implications. Improper disposal of retting water, which contains decomposed plant material, can lead to pollution of water bodies. Therefore, responsible retting practices are essential to minimize environmental impact.

Table 4 The comparison between the natural fiber retting techniques.

Retting method	Process Description	Merits	Limitations	Process duration
Water retting	The process of water retting involves immersing plant stems in water and monitoring them at regular intervals. As the water seeps into the central part of the stem, it causes the inner cells to swell, leading to the rupture of the outermost layer.	▪ Remarkable degree of consistency and excellence in their overall quality. ▪ Resulting fibers are characterized by an even and uniform level of retting throughout their structure.	▪ Associated with significant pollution stemming from anaerobic bacterial fermentation. ▪ Produces foul-smelling odors. ▪ The process is resource intensive.	7-14 days
Dew retting	Plant stalks are evenly distributed across fields, allowing them to be exposed to ample sunlight, atmospheric air, and dew. This exposure facilitates the colonization of fungi, which in turn leads to the decomposition of cellular stem tissues and adhesives.	▪ Minimal water requirement, making it a preferred option in regions facing water scarcity. ▪ Relies on natural atmospheric conditions. ▪ Reduced energy consumption and operational costs.	▪ It relies heavily on weather conditions, making it less predictable and potentially slower than other retting methods. ▪ It is highly susceptible to contamination with soil.	2-10 weeks
Enzymatic retting	Involves the application of specific enzymes to facilitate the breakdown of non-cellulosic components that bind the fibers to the plant material. Enzymes facilitate the breakdown of gum and pectin materials present in the stem through a process known as hydrolysis.	▪ Precise control over the retting process. ▪ Reduced processing time compared to traditional retting methods. ▪ More potential to produce high-quality fibers with specific characteristics	▪ High costs, as enzymes used can be expensive to acquire and apply. ▪ Requires high level of expertise and training for the appropriate selection and implementation of enzymes. ▪ Requires meticulous monitoring to achieve optimal conditions for enzyme activity.	1 -2 days

Retting method	Process Description	Merits	Limitations	Process duration
Chemical retting	Chemical retting is a fiber separation method that involves the use of chemical agents to break down the non-cellulosic components binding plant fibers to the stalks. These chemicals target and degrade the pectins, lignin and other adhesive substances, thus releasing the individual fibers. The plant material is typically immersed in the chemical solution for a specific period, allowing the agents to catalyze the breakdown of the binding materials.	▪ Chemical agents enable manufacturers to finely regulate retting conditions, resulting in consistent and uniform fiber quality. ▪ It is generally faster compared to some natural retting methods, significantly reducing processing times and increasing overall efficiency. ▪ Allows for greater flexibility in adapting to various fiber types, making it applicable to a wide range of plant materials.	▪ Necessitate careful management and treatment to prevent contamination of water sources. ▪ The use of chemicals can lead to the loss of certain properties in the fibers, affecting their color, texture, and overall quality. ▪ The reliance on chemicals also introduces an additional cost factor, as procuring and applying the necessary agents can contribute to higher production expenses.	1-2 hours
Microbial Retting	Microbial retting harnesses the action of microorganisms to break down the non-cellulosic components binding plant fibers to the stalks. The harvested plant material is exposed to a controlled environment conducive to microbial activity. The microorganisms, including bacteria and fungi, produce enzymes that target and degrade pectins, lignin and other adhesive substances present in the plant material thereby loosening the fibers.	▪ Relies on natural processes involving microorganisms rather than chemical agents hence presents a more sustainable approach aligning well with ecological ideals.	▪ Challenging to control consistency as the process relies heavily on factors such as temperature, humidity and the availability of suitable microorganisms. ▪ Microbial retting also results in longer processing times. ▪ The process produces bad odors during the decomposition process.	10-30 days

3.3. Decortication

In situations where fibers are tightly bound to the plant stem, a process called decortication is employed. This process entails the mechanical removal of the non-fibrous outer layers of the plant material to expose the fibers. Historically, hand decortication was the norm and involved pounding the leaves and scraping away the pulp with a knife, but this approach is labour-intensive and time-consuming [30]. Nowadays, mechanical decorticators offer an efficient alternative. These devices utilize rotating wheels with blunt knives to crush and beat the leaves, leaving behind only the fibers. Figure 3 illustrates how mechanical forces are applied to break the bonds between the fiber and its core. Various types of decorticators, such as crushing rollers, ball mills, hammer mills and drop weights, have been used for fiber processing. For high-value applications, additional treatment is often required to reduce unwanted non-cellulosic remains in the mechanically extracted fibers. However, mechanical decortication can disrupt fiber cell wall structures, leading to defects that may negatively impact mechanical properties and composite performance [31]. The extent of such defects depends on factors like previous retting treatments [32]. Nevertheless, some research indicates that high-quality fibers can be obtained through mechanical decortication, especially in modern mills designed to maintain the integrity and length of the fibers by disentangling and aligning them [33]. The performance of a decorticator is influenced by several parameters, and understanding their effects can help improve current industrial decortication processes, even if there's a trade-off between fiber quantity and quality, depending on the chosen parameters and the condition of the natural fiber source.

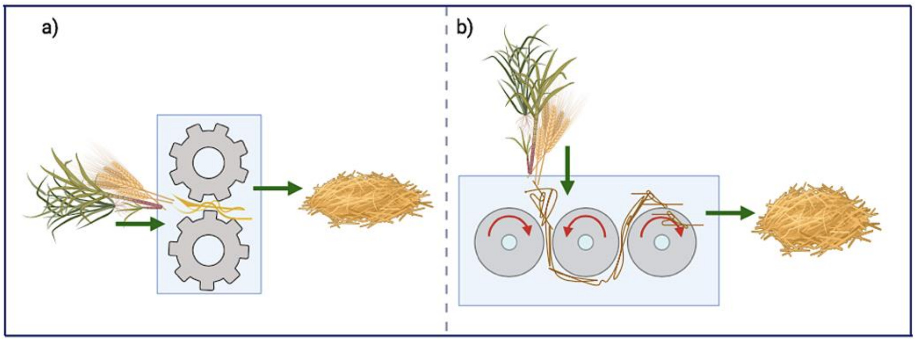

Fig. 3 Illustrations of mechanical decorticators in action; (a)automated decortication by hammering wherein drums with outward-extending hammers that repeatedly strike straw to break it down are employed and (b) automated decortication by roller milling wherein cylindrical rollers are used to break the biomass [36].

Following the initial decortication process, a crucial step known as post-decortication is carried out to refine the fiber extraction. This step involves the separation and cleaning of detached fibers from a mixture containing fiber/core-bound components and fine particles. Various methods are employed for this process. One common method is scutching, which involves further beating or mechanical action to separate the fibers from other components [34]. Another approach involves sieving, utilizing differences in particle size to isolate the fibers. These techniques contribute significantly to the overall quality improvement of the fibers extracted after decortication, making them more suitable for a wide range of applications across industries. Following the extraction of fibers through any of the methods, it is crucial to initiate a comprehensive washing process to remove any residual impurities. Subsequently, the fibers need to undergo a meticulous drying procedure. This is vital because the moisture content within the fibers can significantly impact their overall quality. Notably, artificial drying allows for better control over drying conditions, ensuring that the fibers are dried uniformly and without the risk of overexposure to sunlight, which can lead to bleaching and degradation [35]. In instances where natural drying is chosen, it is important to ensure that the fibers are protected from direct sunlight, they should be dried under shade to prevent them from becoming discolored or weakened due to prolonged exposure to sunlight.

4. NATURAL FIBER CHEMICAL MODIFICATIONS

One of the primary challenges encountered in utilizing natural fibers as reinforcements in polymer composites is the disparity between the hydrophilic nature of the fibers and the hydrophobic nature of the polymer matrix [37]. This incongruity can lead to poor interfacial adhesion and suboptimal composite properties. Addressing this issue involves reducing the inherent hydrophilicity of natural fibers through chemical treatment and surface modification techniques. Recent research endeavors have explored a range of chemical treatment processes targeted at enhancing the surface properties of natural cellulosic fibers. These treatments are aimed at refining the performance of these fibers across diverse applications.

4.1. Mercerization

This treatment procedure involves immersing the fibers in a solution containing strong alkaline substances like sodium hydroxide (NaOH). The mechanism of alkali treatment of a typical cellulose based natural fiber is exemplified in Figure 4. Alkaline agents break down the hemicellulose and lignin components present in the fiber, leaving behind a more refined form of cellulose. This treatment enhances the adhesion between the fiber and the matrix in composite materials through a two-fold process: Firstly, it diminishes the water-attracting characteristics of natural fibers and secondly, it clears away any impurities on the fiber's surface, resulting in a textured surface [38].The enhanced roughness texture of the natural fiber surface from this treatment facilitates a stronger connection between the fibers and the biopolymer matrix, achieved through mechanical interlocking. Another benefit arises from the reduction in the fiber’s hydrophilic nature, which curtails the interaction between the fiber's hydroxyl groups and water molecules [39]. Consequently, issues like fiber swelling, void formation and debonding in biopolymer composites are mitigated. This leads to the production of biopolymer composites exhibiting markedly advanced mechanical properties.

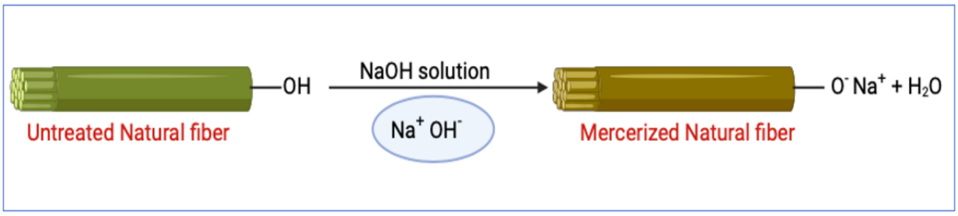

Fig. 4 Illustration of a typical Alkali treatment reaction of cellulose-based fibers with NaOH

Alkali treatment brings about a range of alterations in the characteristics of the natural fibers. The treatment's effects can encompass an elevation in the fiber's tensile strength, elongation at break and stiffness. The degree of these changes can be regulated by factors such as size and morphology of the fibers, soaking duration and concentration of the alkali solution [40]. The duration for which natural fibers are immersed in an alkali solution have been found to be a crucial factor for natural fiber performance. In a study conducted by Setyayunita et al. [41], the structural and functional characteristics of natural fibers exhibited noticeable variations when exposed to varying soaking durations. In the experiment conducted by Zin et al. [42] pineapple leaf fibers were immersed in alkali solution for durations of 1 and 3 hours. Their findings suggested that the most favorable tensile strength was achieved after a 3-hour alkaline treatment. In essence, the duration of the treatment significantly impacts the inherent properties of the fibers, leading to observable distinctions in the resulting behavior and attributes of the composite materials they contribute to.

The concentration of the alkaline treatment has also been identified as a significant factor that profoundly affects both the performance of natural fibers and the properties of the resulting composite materials. Studies have consistently highlighted the substantial impact of the alkaline solution's concentration on the performance of natural fiber performance. Among the notable contributions in concentration effects in mercerization of natural fibers is the work by Boumaaza et al. [43], where the flexural properties of treated natural sisal, flax and jute fibers with respect to varying concentrations of NaOH (1.5%, 2%, and 4%) were investigated. In the study conducted by Cai et al. [44], abaca fibers were subjected to various concentrations (5%, 10%, and 15%) of aqueous NaOH solution. The research highlighted that elevated alkaline concentrations led to reductions in both tensile strength and Young’s moduli as a result of fiber breakage and complete removal of fiber binding materials. Table 5 provides a synopsis of other investigations that have explored the alkali treatment of natural fibers for utilization in composites reinforced by natural fibers.

Table 5 Examples of recent investigations related to alkaline treatments of natural fibers used in natural fiber reinforced composites.

Fiber - matrix	Chemical treatment procedure	Outcome	Ref.
Abutilon indicum – Epoxy (L12)	Initially, the stems were harvested and left to soak in water for approximately 21 days, facilitating the extraction of the fibers. Following this soaking period, the fibers were cleaned and subjected to treatment with a 5% NaOH solution. The alkali-treated fibers then underwent another round of washing using purified water, then dried at 80°C to eliminate any remaining moisture content.	The application of alkali treatment to alter the surface of the fibers led to a notable enhancement in the mechanical properties of the composite samples. Furthermore, the thermal stability of the composite samples treated with alkali exhibited an increase when compared to the untreated composites.	[45]

Fiber - matrix	Chemical treatment procedure	Outcome	Ref.
Dichrostachys Cinerea Bark fibers (DCFs) - Epoxy	The initial process involved immersing the untreated DCFs in a solution of 5% (w/v) NaOH at room temperature. The immersion durations ranged from 30 to 120 minutes. Subsequently, the treated fibers were cleaned thoroughly until the solution was neutralized. Hydrochloric acid solution was added then final rinse with demineralized water was carried out before the fibers are dehydrated in an oven at 80°C for a duration of four hours.	The alkali treatment exhibited a noteworthy impact on the crystalline index of the optimally treated DCFs due to the reduction of amorphous components. Furthermore, the cellulose degradation temperature of the optimally treated DCF improved significantly. Moreover, the mechanical characteristics of the chemically treated samples significantly outperformed those of the untreated raw samples.	[46]
Pineapple leaves - Araldite Epoxy resin	Initially, the pineapple leaves were cleaned and dried for 24 hours in an oven at 70°C. Fibers were then cut into smaller lengths, approximately 2–5 mm. Following this, the fibers were subjected to sodium hydroxide solution of concentrations of 3%, 5%, 7% and 10% for an hour. Thorough cleaning with deionized water was carried out to ensure the removal of any excess alkali chemicals that may have adhered to the surface of the fibers during the treatment.	The alkaline treatment demonstrated a multifaceted enhancement in the fiber's attributes, influencing their strength, thermal behavior and various compositional characteristics.	[47]
Salacca Zalacca fiber (SZF) - High Density Polyethylene (HDPE)	The treatment of SZF involved immersing it in a 5% NaOH solution, with varying immersion times of 1, 2, 3, 4, and 5 hours. The fiber was then washed with 1% acetic acid solution until a neutral pH value was achieved. Subsequently, the SZF was subjected to a rinse with distilled water, after which it was left to air dry at room temperature for approximately 3 days. A further dehydration process was undertaken using an oven set to a temperature of 35°C for 8 hours.	Applying alkali treatment to SZF leads to improvements across several key parameters, including density, crystallinity index, tensile properties and the interfacial shear strength between the fibers and the matrix.	[48]
Chestnut cupula fiber - recycled high-density polyethylene (r-HDPE)	The cupula fibers were carefully dried in a conventional air oven until their moisture content was reduced to less than 1%. Subsequently, the dried cupula fiber underwent treatment with a 5 wt% NaOH solution for around 24 hours at room temperature. After the treatment, the fibers underwent multiple washes with distilled water to ensure the removal of excess alkali. The fibers were once again dried in an oven, this time at 60°C for a period of 24 hours.	The composites consisting of alkali-treated cupula fibers exhibited superior mechanical properties in comparison to their untreated counterparts. This improvement can be attributed to heightened surface roughness and enhanced interface adhesion.	[49]

Fiber - matrix	Chemical treatment procedure	Outcome	Ref.
Lagenaria Siceraria fiber (LSF) – Epoxy	The fibers were washed and subsequently immersed in a solution containing NaOH with a concentration of 4% (w/v) at room temperature. Following this, the fibers were subjected to a rinsing process involving tap water. Afterwards, the fibers were washed with a solution consisting of 1% (v/v) acetone and distilled water. Fibers were then dried in an oven at 60°C for 24 hours.	The compressive, flexural, impact, and tensile strengths of the composite made from 4% alkaline-treated LSF were found to surpass those of the untreated LSF composite. This improvement is attributed to the robust interlocking between the fibers and the matrix.	[50]
Kenaf - Polyvinyl alcohol (PVA)	This treatment was carried out by immersing the fibers in a pre-prepared NaOH solution with concentrations of 4, 6, 8, and 10 weight percentage (wt.%) for a duration of 4 hours, all at room temperature. The fibers were subjected to rinsing using deionized water until a neutral pH was achieved. Treated fibers were then carefully dried in a drying oven set to a temperature of 60°C.	The utilization of NaOH treatment effectively removed impurities, resulting in a cleaner and more textured surface for the Kenaf fibers. Additionally, the treatment led to substantial improvements in the tensile properties of the fibers.	[39]

4.2. Silane treatment

This procedure involves the interaction of the fiber with a silane coupling agent followed by fiber drying and curing stages. The application of silane treatment yields a range of potential modifications on natural fibers. These modifications can involve enhancing the hydrophobic nature of the fibers, along with altering their surface morphology and chemical characteristics. The process of applying silane treatment to natural fibers involves three distinct reactions: hydrolysis, self-condensation, absorption and chemical bonding as illustrated in Figure 5 [51]. Silane molecules undergo hydrolysis when exposed to water and ethanol, releasing alcohol and silanol groups. Subsequently, the silanol groups initiate a self-condensation process, which is deliberately moderated by maintaining an acidic pH environment. This control ensures the presence of smaller molecular-sized free silanol groups. Upon introducing fibers to the silane solution, the hydroxyl groups found in the cellulose/hemicellulose of the fibers absorb the silanol groups through hydrogen bonding. This interaction leads to the formation of robust polysiloxanes (-Si-O-Si-) structures which ultimately establish connections with the matrix through chemical bonding [52].

The degree of modification in natural fibers can be controlled by adjusting key factors: the concentration and duration of the silane treatment, as well as the specific type of silane coupling agent used. This nuanced control has been extensively explored by numerous researchers and scientists, leading to valuable insights. For instance, Youbi et al. [53] conducted a study evaluating the impact of silane treatment on surface modification of Raphia vinifera Fibers (RVF). In their research, fibers were subjected to various concentrations of silane solutions (1% and 5%) to gauge their effects. Alshahrani & Prakash [54] carried out investigations specifically focused on the effects of silane treatment on fibers and their subsequent impacts on composite properties. In their work, raw chopped fibers and peel-dried particles were treated with amino silane using the acid hydrolysis method. In a separate study by Sabarinathan et al. [55], a novel cellulosic fiber extracted from the peduncle portion of the fish tail palm tree underwent treatment with different concentrations (1%, 5%, and 9%) of silane solution. Across these research works, a consistent trend emerged: silane-treated fibers exhibited superior mechanical properties when compared to untreated fibers. Additionally, it was noted that higher concentrations of coupling agents and prolonged treatment durations led to more pronounced modifications in the properties of the fibers. In addition, certain investigations have delved into the impacts of various silane coupling agents. For instance, Wang et al. [56] conducted a study focusing on the mechanical properties and thermal behavior of bamboo fibers reinforced polypropylene composites under the influence of different silane treatments. This study involved the use of three distinct silane coupling agents: γ-Aminopropyltriethoxysilane, γ-Methacryloxypropyltrimethoxysilane and 3-Glycidyloxypropyltriethoxysilane. The research found that the tensile strength and flexural strength of the silane-treated fiber composites displayed enhancements when compared to untreated fiber composites, this was attributed to an enhanced interfacial adhesion between the fibers and the matrix. This study underscored the importance of selecting the appropriate silane coupling agent to achieve desired improvements in the performance characteristics of composite materials.

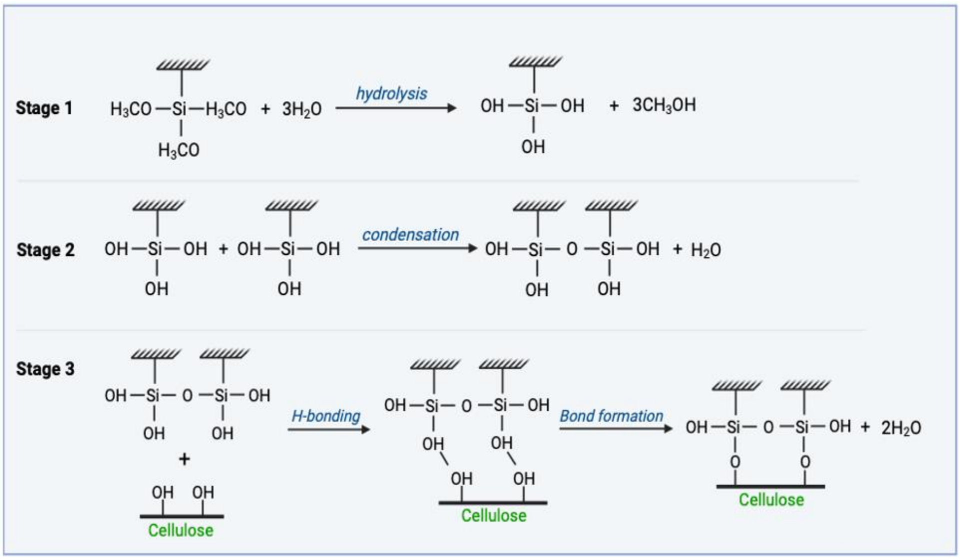

Fig. 5 The general mechanism underlying the formation between silanes (e.g. 3-Glycidyloxypropyl trimethoxysilane shown here), and the surface of the fiber.

4.3. Acetylation

Acetylation, a widely employed chemical treatment, serves as a versatile method for altering the characteristics of natural fibers. This treatment revolves around the interaction of cellulose with an acetylating agent, such as acetic anhydride or acetic acid, usually in the presence or absence of a catalyst as illustrated on Figure 6. The fundamental concept behind acetylation is the substitution of hydroxyl groups (OH) present in natural fibers with acetyl groups (CH3CO), effectively rendering the fiber surface more hydrophobic [57]. The acetylation process can yield a spectrum of modifications to the properties of natural cellulosic fibers, encompassing heightened resistance to water, improved dimensional stability and increased thermal stability. Furthermore, the treatment can induce alterations in the fiber's surface morphology and bring about changes in its chemical and physical attributes. The extent of modification achievable can be fine-tuned by adjusting the concentration and duration of the acetylation treatment. Oladele et al. [58] conducted investigations which revealed that employing higher concentrations of acetylating agents and extending the treatment duration result in more pronounced transformations in the natural fiber's properties. Beztout et al. [59] investigated how acetylation processes and cellulose content affect mechanical, thermal and rheological properties of PCL/cellulose composites. The treatment resulted in improved interfacial adhesion and dispersion of cellulose within the PVC matrix, leading to an enhanced surface morphology. Furthermore, Boustani et al. [60] conducted a comprehensive investigation into the impact of solvent-free acetylation using acetic anhydride on fiber structures. In their study, they acetylated flax and wood pulp fibers at room temperature in a solvent-free system, employing acetic anhydride and sulfuric acid as a catalyst. The results revealed a direct relationship between the degree of fiber acetylation and the reaction duration. Additionally, Souza Da Rosa et al. [61] explored the feasibility of acetylation techniques applied to natural fibers derived from waste materials originating from pulp and paper mill operations. Their study encompassed four distinct acetylation methodologies, broadening the understanding of the potential applications of acetylation across various fiber sources.

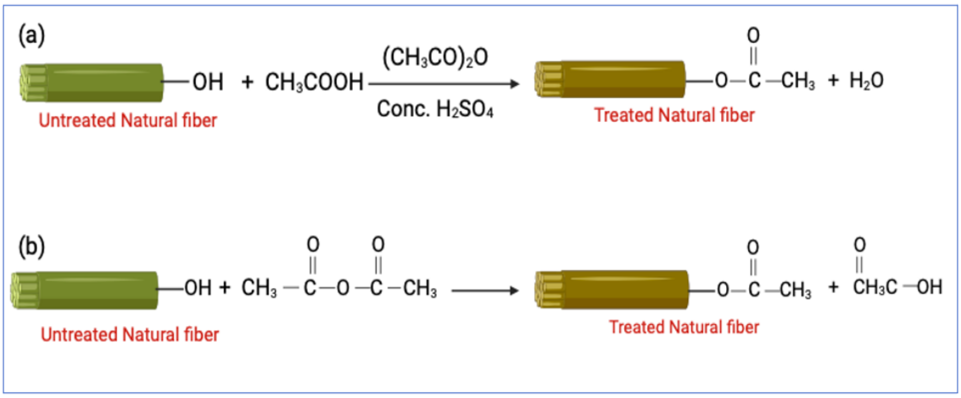

Fig. 6 Acetylation mechanisms, (a) with acid catalyst and (b) without acid catalyst.

4.4. Bleaching

Bleaching is a common chemical treatment employed to alter the characteristics of natural cellulosic fibers like cotton, bamboo and hemp. This treatment involves the use of various chemical agents, including hydrogen peroxide, chlorine dioxide and sodium hypochlorite [62]. The selection of a specific bleaching agent hinges on the unique requirements of the fiber in question and its intended application. Through the bleaching process, natural fibers undergo several alterations, such as increased luminosity, enhanced capacity for dye absorption and improved mechanical attributes. However, if not conducted properly, bleaching can also weaken the fibers. The extent of these modifications is influenced by factors like the type and concentration of the bleaching agent, the temperature, the pH conditions and the duration of treatment [63,64]. However, there are growing concerns regarding the ecological consequences of the chemicals utilized in conventional bleaching procedures. As a result, efforts are underway to develop more sustainable alternatives involving the use eco-friendly bleaching agents and enzymatic bleaching approaches. Many investigative studies in this regard have been explored. For instance, Pereira et al. [65] explored the influence of acetosolv and environmentally friendly bleaching treatment on orange bagasse fiber. Their findings suggest that all the treatment variables employed in their investigation significantly contribute to the fiber's characteristics. The use of this eco-friendly bleaching approach holds promise for employing orange bagasse fibers as reinforcement in polymer matrix applications. In a study conducted by Zhang et al. [66], an innovative and eco-friendly scouring and bleaching method for flax rove was investigated, utilizing supercritical carbon dioxide fluid instead of a traditional water-based medium. In comparison with the conventional water-based approach, this proposed supercritical bleaching process conserves substantial amounts of water and energy, establishing it as an eco-friendlier option for producing flax rove and other materials used in the textile industry. Furthermore, Sojka-Ledakowicz et al. [67] introduced a bleaching method that employs the hydrogen peroxide in the gaseous phase. Their results demonstrated that vaporized hydrogen peroxide-based bleaching is not only environmentally friendly but also operates at lower temperatures, saves energy and generates minimal waste compared to the conventional water- and energy-intensive bleaching process. Regarding the use of enzymatic bleaching Singh et al. [68], proposed the use of crude xylano-pectinolytic enzymes for bleaching ramie fibers intended for textile applications. Their conclusion highlighted the environmental safety of enzymatic bleaching, which efficiently removed non-cellulosic impurities, rendering the fibers more suitable for textile use compared to chemical bleaching. Similarly, Hassan et al. [69] conducted bleaching experiments using protease, xylanase and alkaline protease enzymes to evaluate the effects of enzymatic bio-scouring on the physiochemical and mechanical properties of fiber fabrics. Their enzymatic approach yielded results on par with the conventional chemical method.

4.5. Grafting

Grafting treatment is a surface modification process employed to alter the properties of natural cellulosic fibers through the introduction of polymer chains onto the fiber surface, as illustrated in Figure 7. It is achieved through the chemical bonding of monomers or polymers onto the fiber surface via covalent bonds, resulting in the creation of a polymer layer on the fiber surface [70]. Various techniques, such as radiation-induced grafting, chemical grafting, and enzymatic grafting, can be used for the grafting process. The choice of technique is determined by the specific properties required for the fibers and the intended application. Grafting can bring about diverse alterations in the properties of natural cellulosic fibers, including increased hydrophobicity, enhanced mechanical properties and improved thermal stability. The properties of the grafting layer can be finely tuned by adjusting the type and concentration of monomers or polymers utilized, as well as the reaction conditions. These variables have been thoroughly explored by researchers. In a study conducted by Tataru et al. [71], the radiation grafting procedure of flax natural fibers was investigated. The grafting of epoxidized plant oils onto flax fabrics was found to facilitate the formation of an interphase when cured with an epoxy-amine thermoset matrix.

While the composite's tensile strength exhibited a significant improvement of 10-20% compared to untreated fabric composites, the study highlighted the potential of radiation grafting in enhancing composite properties. Additionally, research by Bao et al. [72] demonstrated the application of enzyme-initiated grafting for the modification of cellulose fibers. Besides its excellent environmental friendliness, this method showcased the ability to graft chains with well-controlled structures and lengths onto cellulosic fiber surfaces. As a result, this approach offers an environmentally friendly and controllable means for grafting, which can be effectively employed in the production of various functional cellulosic materials.

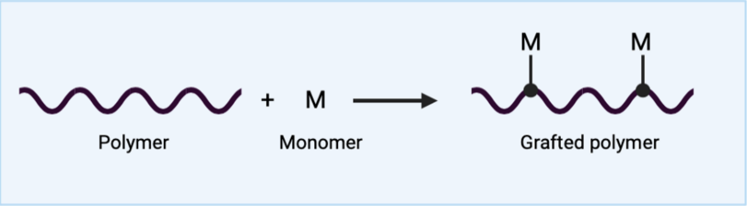

Fig. 7 Illustration of the grafting process [73]

5. SURFACE/PHYSICAL TREATMENT

Physical treatments can also be used to modify the surface of natural cellulosic fibers to improve their compatibility with other materials or enhance their properties. Physical treatments do not alter extensively the chemical composition of the fibers, and therefore the interface is generally enhanced via an increased mechanical bonding between the fiber and the matrix. One the most attractive feature of physical methods is the fact that they are 'clean' since they do not involve chemicals. Some common physical treatment methods are outlined in Table 6.

Table 6 Physical treatment processes for natural fibers.

Surface treatment	Description	Benefits	Drawbacks	Sources
Plasma treatment	Plasma treatment is a process that enhances natural cellulosic fiber's surface properties using low-pressure, non-thermal plasma. This plasma is created by applying an electric field to a low-pressure gas (e.g., oxygen, nitrogen, or argon), generating reactive species. These reactive species interact with the fiber surface, introducing functional groups, surface etching or crosslinking.	▪ Effectively modifies the surface of natural polymers without altering their core properties. ▪ Increases the wettability of the fiber-matrix interface, without causing pollution.	▪ The etching mechanism can lead to surface degradation and creates pits on the fiber surface.	[74–76]

Surface treatment	Description	Benefits	Drawbacks	Sources
Corona treatment	Corona treatment is an electrical process where high voltage is applied near the fiber surface, generating a corona discharge. This discharge produces reactive species like ozone, which introduce polar groups to enhance fiber surface energy and improve adhesion to polymer matrices.	▪ Swiftly and efficiently enhances the bonding between natural fibers and diverse polymer matrices. ▪ Enhances wettability by increasing surface polarity, promoting compatibility between hydrophilic fibers and hydrophobic matrices.	▪ Extended treatment durations could lead to the development of a considerably uneven fiber surface. ▪ Surface ablation and etching from the process diminishes fiber strength.	[77–79]
UV/Ozone treatment	UV/Ozone treatment of natural fibers is a surface modification technique that involves exposing the fibers to ultraviolet (UV) light in the presence of ozone. This process induces photochemical reactions on the fiber's surface, leading to the formation of oxygen-containing functional groups. These newly introduced polar groups enhance the fiber's wettability and adhesion properties, making it more compatible with polymer matrices and other materials in various applications.	▪ Enhanced fiber surface properties by introducing polar functional groups, resulting in improved wettability and enhanced adhesion to polymer matrices. ▪ Highly versatile and can be applied to a wide range of natural fibers, making it adaptable to various industries.	▪ Fibers can be subjected to UV degradation. ▪ The treatment's effectiveness may vary depending on the type of natural fiber. ▪ While the process is environmentally friendly, its energy consumption may be a concern in terms of overall sustainability.	[80,81]
Thermal treatment	Thermal treatment of natural fibers is a surface modification process that involves subjecting the fibers to elevated temperatures in a controlled environment. During this treatment, the fibers are exposed to heat in an inert gas atmosphere. The high temperature induces chemical changes on the fiber's surface, which can include the removal of impurities, moisture and hemicellulose, resulting	▪ Improved thermal stability of the fibers, this is beneficial where fibers may be exposed to elevated temperatures. ▪ Enhanced flame resistance of natural fibers, reducing their flammability and making them less susceptible to catching fire or sustaining combustion. ▪ Increased crystallinity of	▪ If not controlled precisely and subjected to long exposure, natural fibers mechanical properties become compromised. ▪ Heat treatment of the fibers can also make them more brittle and reduce their flexibility, impacting their suitability for certain applications.	[82–84]

	in enhanced fiber purity and increased cellulose content.	natural fibers, higher crystallinity often leads to improved mechanical properties.		
Electron radiation	Electron radiation treatment of natural fibers is a surface modification technique that involves exposing the fibers to high-energy electrons. During this process, the electrons penetrate the fiber's surface, causing molecular changes and enhancing its surface properties. The energy from the electrons initiates chemical reactions on the fiber's surface, leading to the formation of new functional groups and increased wettability.	▪ This treatment is highly controllable, allowing for precise adjustments to achieve desired modifications.	▪ The exposure to irradiation-induced degradation will lower the peak thermal decomposition temperature of materials with high cellulose content.	[85,86]
Dielectric barrier	Dielectric barrier treatment of natural fibers is a surface modification method that employs a high-frequency electrical discharge in a controlled gas environment to enhance the fiber's surface properties. This process involves placing the fibers between two electrodes separated by a dielectric material, creating a barrier that prevents direct electrical discharge contact with the fibers. When high-voltage alternating current is applied, it ionizes the surrounding gas, forming a plasma. Reactive species generated within the plasma chemically modify the fiber surface, introducing polar functional groups and increasing surface energy.	▪ Reduces water absorption, enhances chemical resistance and fosters stronger interfacial bonding, ultimately increasing the overall performance and versatility of natural fibers in a wide range of applications.	▪ It requires specialized equipment and expertise to create and maintain the necessary electrical discharge conditions. ▪ The process may not be suitable for all types of natural fibers, and achieving uniform treatment across large fiber surfaces can be challenging.	[87,88]

6. CONCLUSIONS

In response to the growing global concern for environmental issues, many industrial sectors have increasingly shifted their focus towards adopting sustainable and eco-friendly materials. The use of agro-waste residues as natural fibers for biocomposite development has emerged as a promising alternative, primarily because these resources are inherently renewable, readily available in the natural ecosystem and help address waste management challenges. Despite the advantages these materials offer, they face challenges in commercialization due to issues such as poor compatibility with polymer matrices, limited wettability, high moisture absorption, and reduced durability. These limitations are closely tied to both the nature of the fibers themselves and the extraction and preparation processes involved in transforming them into final products. Therefore, it is crucial to comprehend the impact of fiber extraction methods and fiber modification procedures to tailor fiber properties to specific application requirements. This review provided a comprehensive understanding of the preparation and processing methods of agro-waste residues into suitable natural fibers for composite development. It was highlighted that the selection of suitable harvesting, retting and decortication method plays a crucial role in the physical properties of the fibers. Additionally, it was deliberated that the choice of retting method depends on factors such as climate, water resources, local traditions and desired fiber quality. Furthermore, various chemical treatments approaches used for natural fibers offer a range of properties by chemically altering the chemical composition of the fiber thereby enhancing compatibility with matrices and modifying hydrophilicity. Physical treatment approaches also play a vital role in modifying the surfaces of natural fibers without changing their chemical compositions. Various physical treatment techniques have been assessed to determine their merits and drawbacks in the modification of natural fiber surfaces. Overall, it is important to understand the holistic process of fiber preparation, starting from its origin in agricultural fields to the production of the final fibers or fillers that are essential for effectively incorporating into a sustainable and eco-friendly composite material.

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REFERENCES

1. Singh, M.K., Tewari, R., Zafar, S., Rangappa, S.M., Siengchin, S., 2023, *A comprehensive review of various factors for application feasibility of natural fiber-reinforced polymer composites,* Results in Materials, 17, 100355.
2. Sanjay, M.R., Siengchin, S., Parameswaranpillai, J., Jawaid, M., Pruncu, C.I., Khan, A., 2019, *A comprehensive review of techniques for natural fibers as reinforcement in composites: Preparation, processing and characterization,* Carbohydr Polym, 207, pp. 108–121.
3. Ogah, A.O., Ezeani, O.E., Nwobi, S.C., Ikelle, I.I., 2022, *Physical and Mechanical Properties of Agro-Waste Filled Recycled High Density Polyethylene Biocomposites*, South Asian Res J Eng Tech, 4, 55–62.
4. Kılınç, A.Ç., Durmuşkahya, C., Seydibeyoğlu, M.Ö., 2017, *Natural fibers,* Fiber technology for fiber-reinforced composites, pp. 209–235.
5. Gebai, S.S., Hallal, A.M., Hammoud, M.S., 2018, *Mechanical properties of natural fiber reinforced polymers: emerging research and opportunities, emerging research and opportunities,* IGI Global.
6. Kalia, S., Kaith, B.S., Kaur, I., 2009, *Pretreatments of natural fibers and their application as reinforcing material in polymer composites—a review,* Polym Eng Sci, 49, pp. 1253–1272.

7. Jiménez, L., Rodríguez, A., 2010, *Valorization of agricultural residues by fractionation of their components*, Open Agric J. 4, pp.125-134.
8. Santolini, E., Bovo, M., Barbaresi, A., Torreggiani, D., Tassinari, P., 2021, *Turning agricultural wastes into biomaterials: Assessing the sustainability of scenarios of circular valorization of corn cob in a life-cycle perspective*, Applied Sciences, 11, 6281.
9. Naeem, M.A., Siddiqui, Q., Khan, M.R., Mushtaq, M., Wasim, M., Farooq, A., Naveed, T., Wei, Q., 2022, *Bacterial cellulose-natural fiber composites produced by fibers extracted from banana peel waste*, Journal of Industrial Textiles, 51, pp. 990S-1006S.
10. Morales, M.A., Atencio Martinez, C.L., Maranon, A., Hernandez, C., Michaud, V., Porras, A., 2021, *Development and characterization of rice husk and recycled polypropylene composite filaments for 3D printing*, Polymers (Basel), 13, 1067.
11. Dungani, R., Karina, M., Sulaeman, A., Hermawan, D., Hadiyane, A., 2016, *Agricultural waste fibers towards sustainability and advanced utilization: a review*, Asian J Plant Sci, 15, pp. 42–55.
12. Jayaprakash, K., Osama, A., Rajagopal, R., Goyette, B., Karthikeyan, O.P., 2022, *Agriculture waste biomass repurposed into natural fibers: a circular bioeconomy perspective*, Bioengineering, 9, 296.
13. FAO, 2017, *The future of food and agriculture and challenges*, Food and agriculture organization of the United Nations, Rome.
14. Lokantara, I.P., Suardana, N.P.G., Surata, I.W., Winaya, I.N.S., 2020, *A review on natural fibers: extraction process and properties of grass fibers,* International Journal of Mechanical Engineering and Technology (IJMET), 1, pp. 84–91.
15. Dayan, M.D.A.R., Rahman, M.D.M., Ferdush, J., Kaysar, M.A., Uddin, M.D.M., 2022, *Effect of Harvesting Time on the Fiber Yield and Characterization of Jute Fiber*, Journal of Natural Fibers, 19, pp. 12434–12445.
16. Dudarev, I., 2022, *A review of fibre flax harvesting: conditions, technologies, processes and machines*, Journal of Natural Fibers, 19, pp. 4496–4508.
17. Pari, L., Baraniecki, P., Kaniewski, R., Scarfone, A., 2015, *Harvesting strategies of bast fiber crops in Europe and in China*, Ind Crops Prod, 68, pp. 90–96.
18. Stevens, C.V., 2010, *Industrial applications of natural fibres: structure, properties and technical applications*, John Wiley & Sons.
19. Lee, C.H., Khalina, A., Lee, S., Liu, M., 2020, *A comprehensive review on bast fibre retting process for optimal performance in fibre-reinforced polymer composites*, Advances in Materials Science and Engineering, 2020, pp. 1–27.
20. Mohankumar, D., Amarnath, V., Bhuvaneswari, V., Saran, S.P., Saravanaraj, K., Gogul, M.S., Sridhar, S., Kathiresan, G., Rajeshkumar, L., 2021, *Extraction of plant based natural fibers–A mini review*, IOP conference series: materials science and engineering, 012023.
21. Konczewicz, W., Zimniewska, M., Valera, M.A., 2018, *The selection of a retting method for the extraction of bast fibers as response to challenges in composite reinforcement*, Textile Research Journal, 88, pp. 2104–2119.
22. Ahmed, Z., Rahman, T., Timmi, U.H., Mehrin, S., 2023, *Types, stages and mechanism of action of Jute retting: An overview*, International Journal of Scholarly Research and Reviews, 2, 10.56781/ijsrr.2023.2.2.0026, pp. 009-020
23. Stawski, D., Çalişkan, E., Yilmaz, N.D., Krucińska, I., 2020, *Thermal and mechanical characteristics of okra (abelmoschus esculentus) fibers obtained via water-and dew-retting*, Applied Sciences, 10, 5113.
24. Yu, X., Xia, Y., Liang, D., Fu, W., Yin, C., 2022, *Effect of Warm-Water Retting Pretreatment on the Physical Properties of Banana Stem and Its Fibre*, Materials, 15, 8462.
25. Narkpiban, K., Poonsawat, T., 2022, *Optimizing Cellulose Extraction from Kenaf (Hibiscus Cannabinus L.) Fiber by Selective Retting and Hydrothermal Pretreatment*, Journal of Natural Fibers, 19, pp. 700–713.
26. De Prez, J., Van Vuure, A.W., Ivens, J., Aerts, G., Van de Voorde, I., 2019, *Effect of enzymatic treatment of flax on chemical composition and the extent of fiber separation*, Bioresources, 14, pp. 3012–3030.
27. Rozyanty, A.R., Zhafer, S.F., Shayfull, Z., Nainggolan, I., Musa, L., Zheing, L.T., 2021, *Effect of water and mechanical retting process on mechanical and physical properties of kenaf bast fiber reinforced unsaturated polyester composites*, Compos Struct, 257, 113384.
28. Ruan, P., Raghavan, V., Du, J., Gariepy, Y., Lyew, D., Yang, H., 2020, *Effect of radio frequency pretreatment on enzymatic retting of flax stems and resulting fibers properties*, Ind Crops Prod, 146, 112204.
29. Réquilé, S., Mazian, B., Grégoire, M., Musio, S., Gautreau, M., Nuez, L., Day, A., Thiébeau, P., Philippe, F., Chabbert, B., 2021, *Exploring the dew retting feasibility of hemp in very contrasting European environments: Influence on the tensile mechanical properties of fibres and composites*, Ind Crops Prod, 164, 113337.
30. Hulle, A., Kadole, P, Katkar, P., 2015, *Agave Americana Leaf Fibers*, Fibers, 3, pp. 64–75.
31. Sadrmanesh, V., Chen, Y., Rahman, M., AL-Oqla, F.M., 2019, *Developing a decision making model to identify the most influential parameters affecting mechanical extraction of bast fibers*, J Clean Prod, 238, 117891.

32. Van de Weyenberg, I., Ivens, J., De Coster, A., Kino, B., Baetens, E., Verpoest, I., 2003, *Influence of processing and chemical treatment of flax fibres on their composites,* Compos Sci Technol, 63, pp. 1241–1246.
33. Widyasanti, A., Napitupulu, L.O.B., Thoriq, A., 2020, *Physical and mechanical properties of natural fiber from Sansevieria trifasciata and Agave sisalana*, IOP Conference Series: Earth and Environmental Science, 012032.
34. Miao, M., Pierlot, A.P., Millington, K., Gordon, S.G., Best, A., Clarke, M., 2013, *Biodegradable mulch fabric by surface fibrillation and entanglement of plant fibers*, Textile Research Journal, 83, pp. 1906–1917.
35. Bachchan, A.A., Das, P.P., Chaudhary, V., 2022, *Effect of moisture absorption on the properties of natural fiber reinforced polymer composites: A review*, Mater Today Proc, 49, pp. 3403–3408.
36. Sisti, L., Totaro, G., Vannini, M., Celli, A., 2018, *Retting Process as a Pretreatment of Natural Fibers for the Development of Polymer Composites,* Lignocellulosic Composite Materials, pp. 97–135.
37. Mohammed, M., Jawad, A.J.M., Mohammed, A.M., Oleiwi, J.K., Adam, T., Osman, A.F., Dahham, O.S., Betar, B.O., Gopinath, S.C.B., Jaafar, M., 2023, *Challenges and advancement in water absorption of natural fiber-reinforced polymer composites*, Polym Test, 124, 108083.
38. Kumar, N.P., Chellapandian, M., Arunachelam, N., Vincent, P., 2022, *Effect of mercerization on the chemical characteristics of plant-based natural fibers*, Mater Today Proc, 68, pp. 1201–1207.
39. Samaei, S.E., Mahabadi, H.A., Mousavi, S.M., Khavanin, A., Faridan, M., Taban, E., 2022, *The influence of alkaline treatment on acoustical, morphological, tensile and thermal properties of Kenaf natural fibers*, Journal of Industrial Textiles, 51, pp. 8601S-8625S.
40. Setswalo, K., Molaletsa, N., Oladijo, P., Akinlabi, E., Sanjay, M.R., Siengchin, S., 2021, *The Influence of Fiber Processing and Alkaline Treatment on the Properties of Natural Fiber-reinforced Composites: A Review*, Applied Science and Engineering Progress, 4, pp. 632-650
41. Setyayunita, T., Widyorini, R., Marsoem, S.N., Irawati, D., 2022, *Effect of Different Conditions of Sodium Chloride Treatment on the Characteristics of Kenaf Fiber-Epoxy Composite Board*, Journal of the Korean Wood Science and Technology, 50, pp. 93–103.
42. Zin, M.H., Abdan, K., Mazlan, N., Zainudin, E.S., Liew, K.E., 2018, *The effects of alkali treatment on the mechanical and chemical properties of pineapple leaf fibres (PALF) and adhesion to epoxy resin*, IOP Conference Series: Materials Science and Engineering, 012035.
43. Boumaaza, M., Belaadi, A., Bourchak, M., 2021, *The Effect of Alkaline Treatment on Mechanical Performance of Natural Fibers-reinforced Plaster: Optimization Using RSM, Journal of Natural Fibers*, 18, pp. 2220–2240.
44. Cai, M., Takagi, H., Nakagaito, A.N., Li, Y., Waterhouse, G.I.N., 2016, *Effect of alkali treatment on interfacial bonding in abaca fiber-reinforced composites,* Compos Part A Appl Sci Manuf, 90, pp. 589–597.
45. Mohana Krishnudu, D., Sreeramulu, D., Reddy, P.V., 2020, *Alkali Treatment Effect: Mechanical, Thermal, Morphological, and Spectroscopy Studies on Abutilon Indicum Fiber-Reinforced Composites*, Journal of Natural Fibers, 17, pp. 1775–1784.
46. Baskaran, P.G., Kathiresan, M., Pandiarajan, P., 2022, *Effect of Alkali-treatment on Structural, Thermal, Tensile Properties of Dichrostachys Cinerea Bark Fiber and Its Composites*, Journal of Natural Fibers, 19, pp. 433–449.
47. Jain, J., Sinha, S., Jain, S., 2021, *Compendious Characterization of Chemically Treated Natural Fiber from Pineapple Leaves for Reinforcement in Polymer Composites*, Journal of Natural Fibers, 18, pp. 845–856.
48. Ariawan, D., Rivai, T.S., Surojo, E., Hidayatulloh, S., Akbar, H.I., Prabowo, A.R., 2020, *Effect of alkali treatment of Salacca Zalacca fiber (SZF) on mechanical properties of HDPE composite reinforced with SZF*, Alexandria Engineering Journal, 59, pp. 3981–3989.
49. Boran Torun, S., Pesman, E., Donmez Cavdar, A., 2019, *Effect of alkali treatment on composites made from recycled polyethylene and chestnut cupula*, Polym Compos, 40, pp. 4442–4451.
50. Nagappan, S., Subramani, S.P., Palaniappan, S.K., Mylsamy, B., 2022, *Impact of alkali treatment and fiber length on mechanical properties of new agro waste Lagenaria Siceraria fiber reinforced epoxy composites*, Journal of Natural Fibers, 19, pp. 6853–6864.
51. Sepe, R., Bollino, F., Boccarusso, L., Caputo, F., 2018, *Influence of chemical treatments on mechanical properties of hemp fiber reinforced composites*, Compos B Eng, 133, pp. 210–217.
52. Kenned, J.J., Sankaranarayanasamy, K., Kumar, C.S., 2021, *Chemical, biological, and nanoclay treatments for natural plant fiber-reinforced polymer composites: A review*, Polymers and Polymer Composites, 29, pp. 1011–1038.
53. Youbi, S.B.T., Tagne, N.R.S., Harzallah, O., Huisken, P.W.M., Stanislas, T.T., Njeugna, E., Drean, J.Y., Bistac-Brogly, S., 2022, *Effect of alkali and silane treatments on the surface energy and mechanical performances of Raphia vinifera fibres*, Ind Crops Prod, 190, 115854.
54. Alshahrani, H., Prakash, V.R.A., 2022, *Effect of silane-grafted orange peel biochar and areca fibre on mechanical, thermal conductivity and dielectric properties of epoxy resin composites*, Biomass Convers Biorefin, pp. 1-9.

55. Sabarinathan, P., Rajkumar, K., Annamalai, V.E., Vishal, K., 2020, *Characterization on chemical and mechanical properties of silane treated fish tail palm fibres*, Int J Biol Macromol, 163, pp. 2457–2464.
56. Wang, Q., Zhang, Y., Liang, W., Wang, J., Chen, Y., 2020, *Effect of silane treatment on mechanical properties and thermal behavior of bamboo fibers reinforced polypropylene composites*, J Eng Fiber Fabr, 15, 1558925020958195.
57. Subroto, E., Cahyana, Y., Indiarto, R., Rahmah, T.A., 2023, *Modification of Starches and Flours by Acetylation and Its Dual Modifications: A Review of Impact on Physicochemical Properties and Their Applications*, Polymers (Basel), 15, 2990.
58. Oladele, I.O., Michael, O.S., Adediran, A.A., Balogun, O.P., Ajagbe, F.O., 2020, *Acetylation treatment for the batch processing of natural fibers: Effects on constituents, tensile properties and surface morphology of selected plant stem fibers*, Fibers, 8, 73.
59. Beztout, M., Boukerrou, A., Djidjelli, H., Barres, C., Fenouillot, F., 2015, *Effects of acetylation process and cellulose content on the mechanical, thermal, morphological and rheological properties of poly (vinyl chloride)/cellulose composites*, Cellul. Chem Technol, 49, pp. 517–528.
60. El Boustani, M., Brouillette, F., Lebrun, G., Belfkira, A., 2015, *Solvent-free acetylation of lignocellulosic fibers at room temperature: Effect on fiber structure and surface properties*, J Appl Polym Sci, 132, 42247.
61. Souza Da Rosa, T., Trianoski, R., Michaud, F., Belloncle, C., Iwakiri, S., 2022, *Efficiency of Different Acetylation Methods Applied to Cellulose Fibers Waste from Pulp and Paper Mill Sludge*, Journal of Natural Fibers, 19, pp. 185–198.
62. Vinod, A., Sanjay, M.R., Suchart, S., Jyotishkumar, P., 2020, *Renewable and sustainable biobased materials: An assessment on biofibers, biofilms, biopolymers and biocomposites*, J Clean Prod, 258, 120978.
63. Seisa, K., Chinnasamy, V., Ude, A.U., 2022, *Surface Treatments of Natural Fibres in Fibre Reinforced Composites: A Review*, Fibres & Textiles in Eastern Europe, 30, pp. 82–89.
64. Dursun, S., Yıldız, S.Z., 2023, *Eco-Friendly Bleaching of Cotton Fabrics Without Heating Using Direct Process Water in the Presence of Sodium Chlorite and Phosphonate*, Journal of Natural Fibers, 20, 2146248.
65. Pereira, P.H.F., Santos, L.B.U., Monticeli, F., Cioffi, M.O.H., 2023, *Influence of acetosolv and eco-friendly bleaching treatment of orange bagasse in biocomposite properties: a mechanical and thermal approach*, Cellulose, 30, pp. 3083–3097.
66. Zhang, J., Zheng, H., Zheng, L., 2017, *A novel eco-friendly scouring and bleaching technique of flax rove using supercritical carbon dioxide fluid*, J Eng Fiber Fabr, 12, 155892501701200420.
67. Sojka-Ledakowicz, J., Walawska, A., Filipowska, B., Lewartowska, J., Olczyk, J., Kiwała, M., 2015, *New eco-friendly method of cellulosic product bleaching with simultaneous disinfection,* Fibres & Textiles in Eastern Europe, 23, 3(111), pp. 115-119.
68. Singh, A., Varghese, L.M., Battan, B., Patra, A.K., Mandhan, R.P., Mahajan, R., 2020, *Eco-friendly scouring of ramie fibers using crude xylano-pectinolytic enzymes for textile purpose*, Environmental Science and Pollution Research. 27, pp. 6701–6710.
69. Hassan, M.M., Saifullah, K., 2019, *Effect of Enzymatic Bio-scouring on the Dyeability, Physicochemical, and Mechanical Properties of Jute Fabrics*, Fibers and Polymers, 20, pp. 578–587.
70. Wei, L., McDonald, A.G., 2020, *A review on grafting of biofibers for biocomposites*, Materials, 9, 303.
71. Tataru, G., Guibert, K., Labbé, M., Léger, R., Rouif, S., Coqueret, X., 2020, *Modification of flax fiber fabrics by radiation grafting: Application to epoxy thermosets and potentialities for silicone-natural fibers composites,* Radiation Physics and Chemistry, 170, 108663.
72. Bao, X., Dong, F., Yu, Y., Wang, Q., Wang, P., Fan, X., Yuan, J., 2020, *Green modification of cellulose-based natural materials by HRP-initiated controlled "graft from" polymerization*, Int J Biol Macromol, 164, pp. 1237–1245.
73. Bhattacharya, A., Misra, B.N., 2004, *Grafting: a versatile means to modify polymers: Techniques, factors and applications*, Prog Polym Sci, 29, pp. 767–814.
74. Gupta, U.S., Dhamarikar, M., Dharkar, A., Chaturvedi, S., Kumrawat, A., Giri, N., Tiwari, S., Namdeo, R., 2021, *Plasma modification of natural fiber: A review*, Mater Today Proc, 43, pp. 451–457.
75. Sun, D., 2016, *Surface modification of natural fibers using plasma treatment*, Chapter 2 in Kalia, S. (Ed.), *Biodegradable green composites*, pp. 18–39, doi: 10.1002/9781118911068.ch2
76. Sawangrat, C., Thipchai, P., Kaewapai, K., Jantanasakulwong, K., Suhr, J., Wattanachai, P., Rachtanapun, P., 2023, *Surface Modification and Mechanical Properties Improvement of Bamboo Fibers Using Dielectric Barrier Discharge Plasma Treatment*, Polymers (Basel), 15, 1711.
77. Ramachandran, A., Mavinkere Rangappa, S., Kushvaha, V., Khan, A., Seingchin, S., Dhakal, H.N., 2022, *Modification of Fibers and Matrices in Natural Fiber Reinforced Polymer Composites: A Comprehensive Review*, Macromol Rapid Commun, 43, 2100862.

78. Sathish, S., Prabhu, L., Gokulkumar, S., Karthi, N., Balaji, D., Vigneshkumar, N., 2021, *Extraction, treatment and applications of natural fibers for bio-composites–a critical review*, International Polymer Processing, 36, pp. 114–130.
79. Otàlora Gonzàlez, C.M., Flores, S.K., Basanta, M.F., Gerschenson, L.N., 2020, *Effect of beetroot (Beta vulgaris L. var conditiva) fiber filler and corona treatment on cassava starch films properties*, Food Package Shelf Life, 26, 100605.
80. Kahl, C., Gemmeke, N., Bagnucki, J., Heim, H.P., 2022, *Investigations on fiber–matrix properties of heat-treated and UV-treated regenerated cellulose fibers*, Compos Part A Appl Sci Manuf, 152, 106669.
81. Mahzan, S., Fitri, M., Zaleha, M., 2017, *UV radiation effect towards mechanical properties of Natural Fibre Reinforced Composite material: A Review*, IOP conference series: materials science and engineering, 012021.
82. Yuan, J.M., Feng, Y.R., He, L.P., 2016, *Effect of thermal treatment on properties of ramie fibers*, Polym Degrad Stab, 133, pp. 303–311.
83. Jirawattanasomkul, T., Likitlersuang, S., Wuttiwannasak, N., Ueda, T., Zhang, D., Voravutvityaruk, T., 2020, *Effects of heat treatment on mechanical properties of Jute fiber–reinforced polymer composites for concrete confinement*, Journal of Materials in Civil Engineering, 32, 04020363.
84. Anggraeni, N.D., Latief, A.E., 2023, *Natural fiber heat treatment on composite material*, AIP Conference Proceedings, 2772(1), 030006.
85. Barbosa, A.P., Costa, L.L., Portela, T.G.R., Moura, E.A., Del Mastro, N.L., Satyanarayana, K.G., Monteiro, S.N., 2012, *Effect of electron beam irradiation on the mechanical properties of buriti fiber*, Matéria (Rio de Janeiro), 17, pp. 1135–1143.
86. Le Moigne, N., Sonnier, R., El Hage, R., Rouif, S., 2017, *Radiation-induced modifications in natural fibres and their biocomposites: Opportunities for controlled physico-chemical modification pathways?,* Ind Crops Prod, 109, pp. 199–213.
87. Ferreira, D.P., Cruz, J., Fangueiro, R., 2019, *Chapter 1 - Surface modification of natural fibers in polymer composites*, In: Koronis, G. and Silva, A. (eds.) Green Composites for Automotive Applications, pp. 3–41, Woodhead Publishing.
88. Vander Wielen, L.C., Ragauskas, A.J., 2006, *Dielectric-barrier discharge initiated grafting to enhance fiber charge*, Chem Eng Commun. 193, pp. 683–688.