

# Chemical and physical modifications of the surface of sisal agave fibre used as a reinforcement in epoxy resin – A Review

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## ABSTRACT

A combination of sisal fibers and bio-based epoxy resin offers good potential for producing environmentally friendly bio-composites with improved or equivalent mechanical properties compared to those obtained using 100% synthetic resins. However, the poor interaction between the two materials caused by the different distribution of electrical charge over the atoms joined by the bond in the functional groups of the chemical structure (polarity) necessitates the modification of one of the surfaces of the constituents through various techniques. The paper discusses available literature on several treatments to improve the adhesion between sisal fibers and thermoset epoxy matrices by achieving favorable wettability, mechanical interlocking, and improved interaction through chemical bonding. It is shown that fiber washing in an NaOH solution followed by rinsing and drying is the prevalent chemical treatment. With NaOH treatments, researchers observed cleaner fibers and this promoted better adhesion with the epoxy matrix. Coupling agents such as silane treatments showed an improved resistance to fiber moisture absorption. Thermal treatments affect the fiber's morphology by increasing the crystallinity of the cellulose leading to stiffer composites. It was also observed that the improvement in fiber-matrix adhesion had an adverse effect on the impact strength of the composite.

## 摘要

剑麻纤维和生物基环氧树脂的结合为生产环保生物复合材料提供了良好的潜力，与使用100%合成树脂获得的材料相比，其机械性能得到了改善或相当。然而，由于化学结构（极性）官能团中键连接的原子上电荷分布不同，导致两种材料之间的相互作用较差，因此需要通过各种技术对成分的一个表面进行改性。本文讨论了关于通过实现有利的润湿性、机械互锁和通过化学键合改善相互作用来提高剑麻纤维和热固性环氧树脂基体之间粘附性的几种处理方法的现有文献。研究表明，在NaOH溶液中洗涤纤维，然后漂洗和干燥是最常见的化学处理方法。通过NaOH处理，研究人员观察到更清洁的纤维，这促进了与环氧树脂基质的更好粘附。硅烷处理等偶联剂显示出对纤维吸湿性的改善。热处理通过增加纤维素的结晶度来影响纤维的形态，从而使复合材料更硬。还观察到，纤维基质粘附力的提高对复合材料的冲击强度产生了不利影响。关键词

## KEYWORDS

Review; Composites; Epoxy; natural sisal fibers; natural fiber treatments

## 关键词

审查; 复合材料; 环氧树脂; 天然剑麻纤维; 天然纤维处理

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## Introduction

In today's environmental conscious society, industry still needs to develop new materials for different applications. However, new lightweight materials required by technological innovations must consider various factors one of which is the environment. As a result, green composite materials are gaining popularity and further advances ongoing research to bridge the gap between environmental conscious ideas and practice. One main idea is to use natural fibers as reinforcement in a polymer matrix, aiming to reduce greenhouse gas emissions and landfill material (Ahmad et al. 2019; Ali et al. 2021; Aravindh et al. 2022; Cruz and Fangueiro 2016; Furtos et al. 2021, 2022; Kamboj et al. 2022; Komal et al. 2020; Xue, Tabil, and Panigrahi 2007).

Composites are formed by combining two or more distinct materials – the matrix and the reinforcement. These materials possess different mechanical, physical, thermal, and electrical properties. When combined, the composite material exhibits synergistic behavior, and the resulting mechanical properties of the composite materials are improved when compared with those of the individual constituents. The matrix transfers the load to the fibers and protects them from the environment, whereas the embedded fibers enhance the mechanical properties of the matrix. The reinforcement is the main load-bearing component of the composite, while the matrix binds the fillers or fibers together.

Composite materials have significant applicability in different market areas, such as transportation, construction, renewable energy harvesting, and aerospace. In general, composites have the potential to replace steel in the automobile industry where the total weight of a vehicle can be reduced by approximately 25% and consumption of nearly 250 million barrels of oil can be saved worldwide (Wazeer et al. 2023). Another example is the percentage of composite material used in airplane manufacture, which increased from 1% in the Boeing 747 of 1979 to 50% in the Boeing 787 of 2011, enhancing the efficiency of the aircraft and saving fuel (Fiore et al. 2016; Shesan et al. 2019).

## The matrix

Composites can be fabricated using a ceramic, metal, or polymer matrix. The word polymer is derived from the Latin word where “Poly” meaning “many” and “Mer” meaning “repeated unit.” A polymer is a large molecule (macromolecule) composed of repeating structural units typically connected by covalent chemical bonds. In industry, it is possible to find thermoplastic and thermosetting polymers. These different types of polymers behave differently when subjected to heat. Thermoplastics soften and eventually melt when heated and can so be recycled. Examples of thermoplastics are polyethylene terephthalate (PET), high- or low-density polyethylene (HDPE, LDPE), polypropylene (PP), polyether ether ketone (PEEK), etc. On the other hand, thermosets retain their form and rigidity when heated and so cannot be remolded. An example of a thermoset is epoxy resin, which has an outstanding chemical and humidity resistivity and thermal resilience (Becker et al. 2012; Behera et al. 2022; Wang et al. 2019).

The source and biodegradability characteristics of a polymer are important factors to assess the sustainability and environmental impact when used in composites. Bio-resins is a common term which can either mean that the raw materials of the resin are sourced from natural renewable resources, usually commercially referred to as “bio-based” resin, and/or the polymer is biodegradable with low or no adverse environmental impact at its end-of-life. According to (Gurunathan, Mohanty, and Sanjay 2015), the latter type of polymers are divided into two categories, those having natural origin and synthetic polymers. The use of bio-polymers on an industrial scale started in the mid-nineteenth century and predates their synthetic counterparts by almost a century when the world was pushed research into the mass production of synthetic polymers (Manu et al. 2022). Nowadays, biopolymers are gaining popularity mainly fueled by sustainability and environmental concerns inherent in petroleum-based polymers.

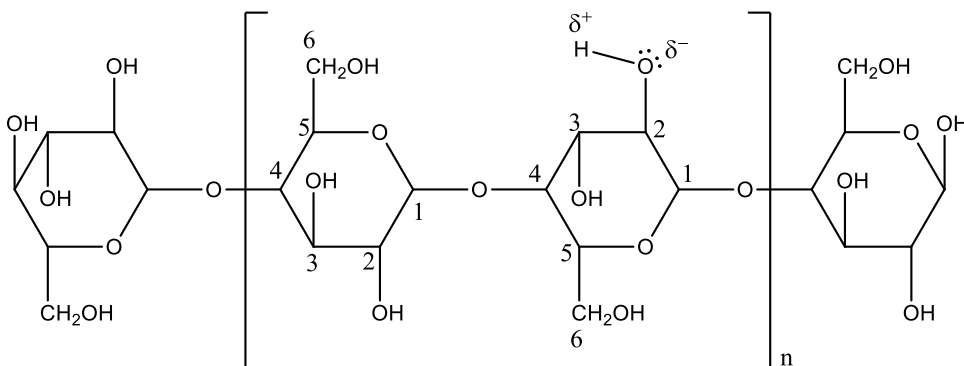
## Natural fibers

Composites composed of fibers sourced from renewable resources and/or are biodegradable, are usually referred to as “green composites,” “bio-composites” or “eco-composites” since the fibers are considered eco-friendly. Natural fibers are obtained directly from plant, animal or mineral resources. Cotton, kenaf, banana, bamboo and sisal are among the most common sources of natural fibers (Ahmad et al. 2019). Unlike synthetic fibers, natural fibers can be composted or incinerated. Natural fibers possess remarkable characteristics, making them applicable to several different areas, due to their biodegradability, low density, recyclability, high specific strength, and modulus, low cost of extraction, and their abundance in nature (Koohestani et al. 2019; Rong et al. 2001; Syduzzaman et al. 2020; Tragoonwichian, Yanumet, and Ishida 2007; Varghese and Mittal 2017). Natural fibers exhibit excellent physicochemical and mechanical properties, almost similar properties to those of synthetic petroleum-based fibers. Table 1 lists the physical and mechanical properties of some notable plant fibers. However, natural fibers have some disadvantages, such as easy degradation and non-consistent mechanical properties; this is as evidenced in Table 1, low thermal stability, moisture absorbance, and hydrophilic properties resulting in poor adhesion with the polymer matrix.

Sound knowledge of the chemical composition of natural fibers is essential to enhance the fiber/resin compatibility. Fiber composition depends on the growing condition, the extraction process, climate, plant age, and natural environment (Aravindh et al. 2022). Fibers consist of cellulose (40–75%), hemicellulose (10–25%), and lignin (515%) which all have complex structures. The cellulose compound is embedded in soft lignin, and the lignin gives additional strength to the fiber and acts as a protection against biological attacks, whereas hemicellulose provides cementing material in the cell wall and forms the auxiliary layer of the fiber network (Agrawal et al. 2000; Aravindh et al. 2022; Kalia et al. 2009). Figure 1 shows a representation of the chemical structure of

**Table 1.** List of typical physical and mechanical properties of some notable plant fibers compiled from several sources (Barbero 2011; Dittenber and GangaRao 2012; Djafari Petroudy 2017; Faruk et al. 2012; Ishak et al. 2010; Suardi et al. 2018; Suherman et al. 2019; Vijayalakshmi, Ch Neeraja, and Hayavadana 2014).

Natural plant fiber	Specific gravity	Diameter	Young's Modulus	Tensile strength	Elongation at break	Cellulose	Lignin
		$\mu\text{m}$	GPa	MPa	%	wt%	wt%
Flax	1.4 – 1.5	12 – 600	27.6 – 103	343 – 2000	1.2 – 3.3	71 – 78	2.2
Sisal	1.33 – 1.5	8 – 200	9.0 – 38	363 – 700	2.0 – 7.0	47 – 78	7 – 11
Abaca	1.50	54 – 260	6.2 – 41	400 – 980	1.0 – 10	53 – 63	7 – 9
Cotton	1.51	10 – 45	12	287 – 597	3 – 10	85 – 90	0.7 – 1.6
Banana	1.35	12 – 30	12 – 29	500 – 914	1.5 – 9.0	63 – 64	5
Bamboo	0.6 – 1.1	25 – 40	11 – 17	140 – 441	1.3 – 8	26 – 43	1 – 31
Hemp	1.4 – 1.5	25 – 500	23.5 – 90	270 – 900	1.0 – 3.5	70 – 74	3.7 – 5.7
Jute	1.3 – 1.45	25 – 200	10 – 30	393 – 800	1.16 – 1.8	61 – 72	12 – 13
Kenaf	1.2 – 1.4	17 – 30	14.5 – 53	223 – 930	1.5 – 2.7	31 – 57	15 – 19



**Figure 1.** Representation of cellulose structure.  $\delta^+$ : partial positive charge;  $\delta^-$ : partial negative charge.

the cellulose, which is the principal fraction of fiber. Cellulose is a semicrystalline polysaccharide consisting of D-anhydrous glucose ( $C_6H_{11}O_5$ ) with  $\beta$ -1,4-glycoside bonding at the C1 and C4 position. Hemicellulose is tightly bound to cellulose fibrils by hydrogen bonding and has a more amorphous structure than cellulose and branched polymers having weaker bonds with regard to strength (Symington et al. 2009). Hemicellulose is hygroscopic, highly hydrophilic, and partially soluble in water due to its open structure, mainly on hydroxyl and acetyl groups. The hemicellulose chains are usually located at the interface between cellulose and lignin, acting as a compatibilizer between both. In the case of lignin, it has the lowest water absorption of the fiber constituents. Lignin possesses a complex and amorphous structure based on aromatic groups, and to date, there are no methods to isolate the lignin in its native state from the fiber. Both materials are responsible for the degradation of the fiber (Ahmad, Hamid, and Osman 2019; Cruz and Figueiro 2016; Sathishkumar et al. 2013; Syduzzaman et al. 2020; Symington et al. 2009).

### ***Agave sisalana***

Agave plants can be grown in harsh conditions such as arid and semi-arid climates. The plants have different taxonomy and species, and some of them can be used for fiber extraction, including the Agave Americana, Agave Americana Variegata mediopicta alba, Agave Americana Variegata Marginata and Agave Sisalana. The latter is widely known as sisal, where the majority originates from Brazil and Tanzania (Li, Mai, and Ye 2000) and can produce substances with antimicrobial, anti-inflammatory and parasiticide properties (Cruz-Magalhães et al. 2019) together with cellulose-rich fibers. Agave is straightforward to cultivate, with a relatively short plantation time. It has approximately a 10-year life-span after which a mature plant can produce about 2000–2500 commercially usable leaves, and each leaf contains around 1000 fibers. Sisal possesses high cellulose content (60–78%) (Dicker et al. 2014; Ferreira, Cruz, and Figueiro 2019; Naveen et al. 2019; Rana et al. 2017; Sahu and Gupta 2017; Satyanarayana, Arizaga, and Wypych 2009; Senthilkumar et al. 2018), and high tensile strength around 468–640 MPa, Young's modulus between 9.4 and 22 GPa, elongation at break between 3% and 7%, a density of  $1.45 \text{ g/cm}^3$  and a diameter between 50 and 200  $\mu\text{m}$  (Gañan et al. 2005; Gebretsadik et al. 2023; Kalia et al. 2009; Senthilkumar et al. 2018).

Gebretsadik et al. (2023) conducted a comparative study of agave americana and agave sisalana leaf fibers as potential alternatives to synthetic fibers. The characterization of the fibers revealed a hydrophilic nature which a higher amount of hemicellulose (17.4%) and moisture (close to 8%). Agave sisalana exhibited a larger amount of cellulose (66.4%) and lignin (16%) compared to agave americana. This was further confirmed by scanning electron microscopy (SEM) which showed a microstructure with a rough surface with numerous cracks. However, the tensile strength (512 MPa) and elongation at break (6%) suggested that the fibers have potential use as reinforcement for composites (Gebretsadik et al. 2023).

When compared with other natural fibers, sisal fibers have inherent toughness and durability which grant them exceptional impact resistance, essential for use in agricultural twines and industrial ropes (Joseph et al. 1999, Naveen et al. 2019). Sisal fibers are also popular for their abrasion resistance, which extends their lifespan in applications where they are subjected to friction, such as in carpets, mats, and various geotextiles (Thomas et al. 2011). Good thermal stability is another property of sisal fibers, allowing them to perform well in environments with varying temperatures thus making them suitable for automotive and construction applications where thermal resistance is necessary (Thomas et al. 2011). Although sisal fibers can absorb moisture, they do not easily degrade in wet conditions. This moisture management capability makes them suitable for agricultural and marine applications where exposure to water is common (Saxena et al. 2011).

The annual global production of sisal fibers in 2023 was approximately 300,000 tons annually (Townsend 2024). The primary exporters include Brazil, Tanzania, and Kenya, with Brazil leading at about 45,000 tons, followed by Tanzania and Kenya producing around 36,000 and 23,000 tons, respectively (Reimer-Wollenweber 2024). The production volume of Agave derived fibers ranks

fifth in the global plant-derived fibers thus underscoring the sisal's significance in various industrial applications, from ropes and twines to composites for automobiles and construction materials (Townsend 2024).

### **The fiber-matrix interface**

In nature, cellulose chains are ordered in a way that allows them to form compact microfibrils through intramolecular hydrogen bonds. Hydrogen bonds and Van der Waals bonds are responsible for the alignment of cellulose macromolecules. Consequently, cellulose chains form highly ordered structures (crystalline regions). In addition, hydrogen bonds make these crystalline structures insoluble in water and most organic solvents. The crystalline cellulose increases the rigidity thus improving the mechanical properties of the fiber. Cellulose is hydrophilic, with hydroxyl groups in each unit available to form hydrogen bonds which could be inter or intra-molecular (Figure 1). This contributes to the cellulose chain to be stiffer and enhances its rigidity (Bisanda 2000; Kocak, Merdan, and Nayci Duman 2016; Sathiamurthi et al. 2021; Syduzzaman et al. 2020).

Due to the presence of hydroxyl groups in cellulose, natural fiber exhibits polar behavior. The bond between the hydrogen and the oxygen in the hydroxyl groups is electrically inequivalent. The hydrogen is slightly positively charged ( $\delta+$ ), and the oxygen is slightly negatively charged ( $\delta-$ ) (Figure 1). This polarity arises due to the electronegativity of the oxygen, which attracts the electrons toward itself, leading to a polar bond. The lone pair electrons of oxygen atoms also affect the electron density in the electron cloud, contributing to the polar nature of the hydroxyl group. As a result of the uneven distribution of the electrical charge, cellulose structure has a polar nature. On the other hand, the natural fibers have hydrophilic properties (while the matrix has hydrophobic properties). This can decrease adhesion between the fiber and resin matrix at the interface and results in poor resistance to moisture absorption due to differences in surface energy. Natural fibers exhibit poor compatibility with nonpolar polymer matrices and low wettability with thermoset matrices. Poor adhesion can cause cracks to develop in the composite when the fibers inside the matrix try to absorb moisture/water, breaking the hydrogen bonds between the fiber and the matrix or between the fibers themselves. This can lead to a high moisture and water absorption which causes swelling and which deteriorates the mechanical strength, therefore shortening the lifespan of the composite material. The ability to transfer the stress through the polymer matrix onto the fibers is therefore drastically reduced, leading to a significant decrease in the composite's matrix/fiber mechanical properties (Ahmad et al. 2019; Cruz and Fanguero 2016; Gañan et al. 2005; Rajkumar et al. 2018; Xue, Tabil, and Panigrahi 2007). Over the years, various methods have been explored to enhance the adhesion between hydrophilic fibers and hydrophobic matrices with the main aim of reducing moisture absorption.

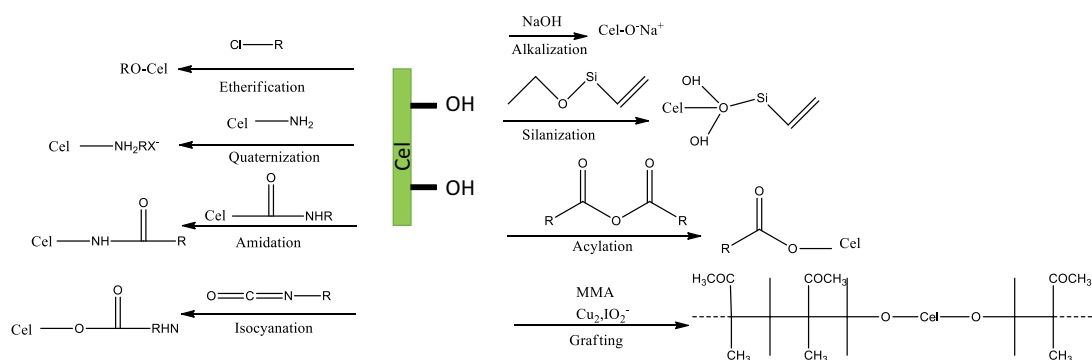
### **Fiber surface modification**

Surface modification is aimed at enhancing compatibility with the matrix, improving surface tension, interfacial strength, and wettability, altering the crystallinity and composition of the fiber, removing impurities from the fiber surface, and reducing the polar nature of the fiber (Ahmad et al. 2019; Aravindh et al. 2022; Senthilkumar et al. 2018). As can be seen in Table 2 and Figure 2, there are different physical, chemical, and combined techniques that can be employed to modify the fiber surface.

- **Physical modification** is used to alter surface properties without modifying the chemical structures, through techniques such as thermal-treatments, electronic discharge from corona and plasma processes.
- **Chemical modification** aims to increase the interfacial bonding between the fiber matrix, by breaking the hydrophilic properties of the fiber, activating the hydroxyl groups, or facilitating interlocking with the matrix due to the introduction of a third component. These groups are double-function coupling agents able to react with hydroxyl groups from cellulose and matrix's

**Table 2.** Fiber modification methods adapted from (Ahmad et al. 2019; Kalia et al. 2009; Xue, Tabil, and Panigrahi 2007).

Fiber modification treatments	
Physical	Chemical
<ul style="list-style-type: none"> <li>• Mechanical               <ul style="list-style-type: none"> <li>Rolling</li> <li>Calendering</li> <li>Stretching</li> <li>Fiber Beating</li> </ul> </li> <li>• Solvent extraction</li> <li>• Electric discharge               <ul style="list-style-type: none"> <li>Corona</li> <li>Plasma</li> <li>Dielectric barrier</li> <li>Ultraviolet</li> <li>Ionized</li> <li>Fiber beating</li> </ul> </li> <li>• Thermal</li> </ul>	<ul style="list-style-type: none"> <li>• Mercerization</li> <li>• Coupling agent               <ul style="list-style-type: none"> <li>Silane</li> <li>Acylation</li> <li>Graft Copolymerization</li> </ul> </li> <li>• Benzoylation</li> <li>• Maleated coupling agents</li> <li>• Acrylation and acrylonitrile Grafting</li> <li>• Isocyanate</li> <li>• Stearic acid</li> <li>• Sodium Chloride</li> <li>• Bleaching               <ul style="list-style-type: none"> <li>Oxidative</li> <li>Reductive</li> </ul> </li> <li>• Enzyme</li> </ul>

**Figure 2.** Chemical modification of plant-based fiber.

functional groups. The most discussed chemical treatments are alkaline, anhydride, silane, acetylene, coupling, and benzoylation. Other techniques like peroxide,  $\text{KMnO}_4$ , baking soda, and ultrasonication have also been explored but to a lesser degree (Ahmad, Hamid, and Osman 2019; Jiang et al. 2021; Komal et al. 2020; Syduzzaman et al. 2020).

There are numerous publications that discuss surface modification practices of reinforcing fibers embedded in a resin matrix as evidenced in numerous review papers such as (Ahmad et al. 2019; Amiandamhen, Meincken, and Tyhoda 2020; Cruz and Fangueiro 2016; U. S. Gupta et al. 2021; Gurunathan, Mohanty, and Sanjay 2015; Jiang et al. 2021; Kenned, Sankaranarayanan, and Suresh Kumar 2021; Komal et al. 2020; Mukhopadhyay and Fangueiro 2009; Saheb and Jog 1999; Syduzzaman et al. 2020; Venkatachalam et al. 2016). This review paper concentrates specifically on studies focused on sisal fiber surface modification carried out to improve the adhesion with polymeric matrices with an emphasis on epoxy resins where available.

### Surface treatments of agave sisalana fibers

Strengthening the epoxy-sisal fiber adhesion requires improving the wettability of the fibers, increasing the mechanical interlocking mechanism and promoting interaction by chemical bonding (Rajkumar et al. 2018). The surface treatments presented in this review are categorized between physical and chemical in nature.

## Physical treatments

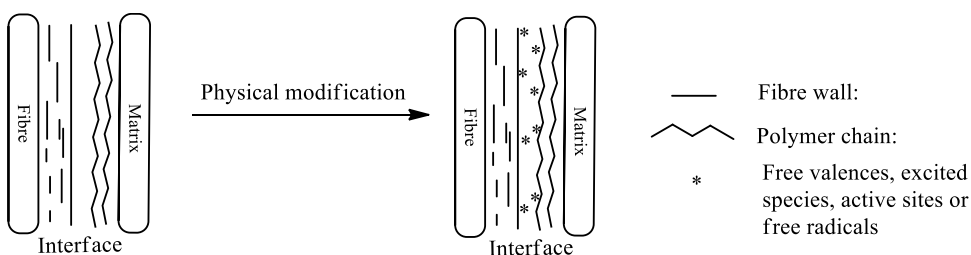
Physical treatments are applied to partially modify the natural fiber surface, morphology, and structural properties without altering the chemical composition and characteristics of the fibers' constituents. The main aim of physical treatments is to cause the fiber to split into fibrils, thus increasing the fiber surface area. This effect increases the compatibility between the reinforcement and the polymer matrix by increasing the fiber roughness or the polar groups formed on the surface. In addition to the formation of fibrils, the fiber treatment shall be capable to release and remove impurities. Physical fiber treatments can be split broadly into three categories: mechanical processes, electric discharge treatments, solvent extraction methods and hydrothermal treatments (Ahmad et al. 2019; Fuqua, Huo, and Ulven 2012; Gurunathan, Mohanty, and Sanjay 2015).

**Mechanical treatments** include stretching, calendaring and rolling where the fibers are aligned and stretched during these purely mechanical processes. Kim and Netravali (2010) observed that applying a certain level of tensile load on the sisal fibers during the mercerization process improved the fiber fracture stress and increased the stiffness which was partially attributed to the lower microfibrillar angle with respect to the fiber axis. This change in microfibrillar angle is brought about by the stretching of the fibers.

**Electric discharge methods** encapsulate various methods such as plasma treatments using different mediums (Gupta et al. 2021; Gupta, Tiwari, and Sharma 2023a, 2023b; U. S. Gupta and Tiwari 2022; Upendra Sharan; Mukhopadhyay, Pal Narula, and Mayank 2013; da Silva et al. 2020; Valášek, Müller, and Šleger 2017; Yuan, Jayaraman, and Bhattacharyya 2002, 2004), ionized air (de Paiva, Maria, and Frollini 2006), ultraviolet radiation, corona discharges (Cruz and Figueiro 2016; Zhou, Fan, and Chen 2016), steam explosion (He et al. 2014), electron radiation, dielectric barrier, gamma radiation and fiber refining.

Electric discharge methods are used to improve the bonding between the fiber and the matrix through the change of the surface energy of the fiber, such as by producing free valences, excited species, active sites, free radicals, or carbonyl groups on the fiber surface (Figure 3). These changes can occur due to bonding scission or hydrogen and hydroxyl abstraction. Treatments such as corona discharges have been expansively used in natural fiber composites, where the electrical discharge leads to produce carbonyl groups activating the surface (Cruz and Figueiro 2016; Zhou, Fan, and Chen 2016). Some physical methods lead to produce opposite charges in both the fiber and the matrix, which results in electrostatic adhesion in the composite interface. As a result, the surface properties are changed and thus improve the bonding between the fiber and the polymer (Amiandamhen, Meincken, and Tyhoda 2020; Aravindh et al. 2022; Zhou, Fan, and Chen 2016).

Plasma treatments act on the surface of the fibers and the bulk properties of the fibers are conserved (da Silva et al. 2020; Sun 2016). Plasma discharges using argon, air, oxygen or nitrogen to surface treat sisal fibers were explored by various researchers. In particular, U. S. Gupta and Tiwari (2022) reported an increase of about 50% in interlaminar shear strength, flexural strength, and elongation at the break of unidirectional sisal-epoxy composites. In addition, an increase of approximately 31% was reported in the tensile strength of the



**Figure 3.** Schematic representation of the fiber-matrix interfacial bonding after physical modification. Straight line: fiber wall. Zigzag line: polymer chain. (\*): free valences, excited species, active sites, free radicals, or carbonyl groups.

composites (U. S. Gupta and Tiwari 2022). An increase in fiber surface roughness was also reported by Yuan, Jayaraman, and Bhattacharyya (2002) where the fibers were exposed to argon or air plasma treatment and then embedded in a polypropylene matrix. The authors reported that the latter treatment resulted in a better interfacial shear strength over the argon-plasma treatment. Furthermore, the moduli and strengths of both tensile and flexural modes improved using either argon- or air-plasma, where the latter showed better improvements (Yuan, Jayaraman, and Bhattacharyya 2004). The results obtained by Valášek, Müller, and Šleger (2017), who treated sisal fibers with oxygen-plasma, also show an increase in fiber surface roughness, although no significant change in tensile strength was reported between the untreated and treated sisal fibers. This supports the claim that plasma treatment does not alter the bulk properties of the fibers.

Ionized-air treatment of short sisal fibers was employed by de Paiva, Maria, and Frollini (2006) where, through SEM imaging and a fixed exposure time of 1 h, it was shown that mercerization and esterification showed better interfacial properties with the phenol-formaldehyde matrix. The authors suggest that varying the exposure time might yield better results for the ionized-air treatment of sisal fibers.

Steam explosion is one of the most efficient and eco-friendly method of treating natural cellulosic fibers (Ziegler-Devin, Chruscziel, and Brosse 2021) where sudden steam decompression causes the constituents of the fibers to physically breakdown. He et al. (2014) treated short sisal fibers using steam explosion and reported that the fibers disintegrated into small fibers thus increasing the surface area. The walls of the newly exposed surfaces consisted of intertwined thin microfibrils, thus increasing further the interfacial area between the fibers and the matrix (He et al. 2014).

**Solvent extraction** is a mechanical fractionation method using selective solvent action where the fibers are selectively separated from the plant sources using chemical solvents. Although this method is perhaps the simplest to apply, the selection of the solvent is not simple since it can degrade the fibers' aspect ratio. In addition, some solvents are not eco-friendly hence the use of such solvents will defy the purpose of using natural fibers (Ahmad et al. 2019; Fuqua, Huo, and Ulven 2012).

**Thermal and hydrothermal** treatments have been explored as physical treatments to accelerate the aging process of natural fibers. These tests have the objective of evaluating the changes in the properties of natural fibers due to environmental effects. The elevated temperature applied to the material may increase the crystallinity of the hard cellulose due to adjustment in the molecular structure of the fiber. As a result, it can lead to higher fiber stiffness and extract a portion of hemicellulose, reducing moisture absorbance. The work done by Rong et al. (2001, 2002) showed that heating treatment improves the sisal-epoxy resin interface by increasing the surface area in contact with the matrix, thereby enhancing fiber wettability (Kalia et al. 2009; Meenakshi and Krishnamoorthy 2019).

Kamboj et al. (2022) investigated pre-treatment hornification methods of agave-fiber reinforced epoxy composites under hygrothermal aging conditions. The alkali hornification method has the potential to reduce the mass gain of treated agave fiber composites by at least 30% and up to 120% when compared with untreated fiber composites, demonstrating higher resistance to moisture absorption. According to the SEM analysis, the shrinkage of the fiber cells after thermal treatment may increase surface roughness, thereby improving bond with the matrix (Kamboj et al. 2022).

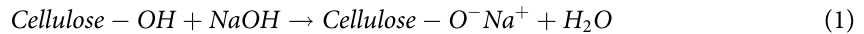
### **Chemical treatments**

The hydrophilic nature of the fibers poses a challenge in manufacturing fiber-polymer composite. Chemical pre-treatment of the fiber is necessary to alter it chemically and to overcome the poor interfacial adhesion between the polar-hydrophilic fiber and nonpolar-hydrophobic matrix. The use of chemical processes for fiber surface modification involves treating the polarized hydroxyl group by modifying the chemical structure of the fiber to reduce its polar nature.



### Alkali treatment

One common example of an alkali treatment is the mercerization process, which treats the OH group of the cellulose as shown in equation (1). The use of chemical compounds aims to improve the adhesion between the fiber surface and the polymer matrix and increase fiber strength. According to Rajkumar et al. (2018), surface energy can be used to evaluate the enhancement in the composite compatibility, which can influence the performance of the fiber reinforced composite. Chemical treatment increases the fiber surface energy, improving its adhesion to the matrix and yielding better fiber-matrix performance. The modification is made through the immersion of the fibers into chemical solutions (Ahmad et al. 2019; Cruz and Figueiro 2016; Rajkumar et al. 2018; Xue, Tabil, and Panigrahi 2007).



Numerous research papers have been published about the use of the mercerization method as being one of the most important methods in modifying the adhesion properties of the fiber-reinforced polymer matrix. In this process, the fiber is immersed in an alkaline solution, and the native crystalline cellulose-I is converted to more thermodynamic favorable cellulose-II. The desired changes in crystallinity in plant fibers can be difficult to achieve. The effects of reaction time, chemical concentration and temperature have been some of the parameters studied in the literature. Materials as NaOH break the bonds of the hydrophilic groups (OH), which dissolves the lignin and cellulose (Fortea-Verdejo et al. 2017).

The alkaline process is a low-cost and easy-to-use methodology that promotes the ionization of hydroxyl groups to alkoxides and disrupts the hydrogen bonding in the fiber network structure, thus increasing the fiber reactivity (equ. 1). As a result, the treatment makes the fiber surface more porous, increasing the chances for the resin to penetrate the fiber by capillary action and filling the gaps (Akash et al. 2016; Rajkumar et al. 2018) (Figure 4). Mercerization such as the alkaline treatment also leads to fibrillation, which breaks down the original fiber into slender ones, increases the roughness of the surface texture, reduces the fiber diameter, and increases the potential reaction sites. At the same time, the alkaline treatment exposes an amount of cellulose and short-length crystallites due to the partial or total removal of some impurity fraction and some polar groups from the fiber surface and some hydrogen bonding in the network structure (Kalia et al. 2009). As a result, the resin is allowed to penetrate into the created voids within the fiber during the composite curing process, leading to the formation of a mechanical network where the fibers interlock with the matrix (Figure 4) (Tragoonwichian, Yanumet, and Ishida 2007). The alkaline treatment is also capable of removing pectin, wax, oils, and amorphous components, dissolving the lignin and hemicellulose that cover the exterior fiber surface cell walls. Figure 5 shows SEM micrographs of sisal fibers before and after NaOH treatment at 2 wt% concentration. The treatment was carried out by the authors to qualitatively illustrate the cleaning effect of NaOH on sisal fibers. By comparing the two micrographs in Figure 5, it is clear that before the alkaline treatment, the surface showed the presence of random particles (shown as white areas in Figure 5a) on the fibers' surface, which are related to wax, lignin, hemicellulose, silica, pectin, and other impurities which was also noted by (Reddy et al. 2013) on the Agave American species.

Moreover, alkaline treatment results in an increase in the amount of cellulose (crystalline) material on the fiber surface, lower moisture absorption capacity, and minimized thermal and biological

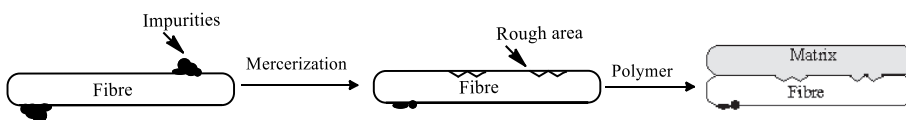
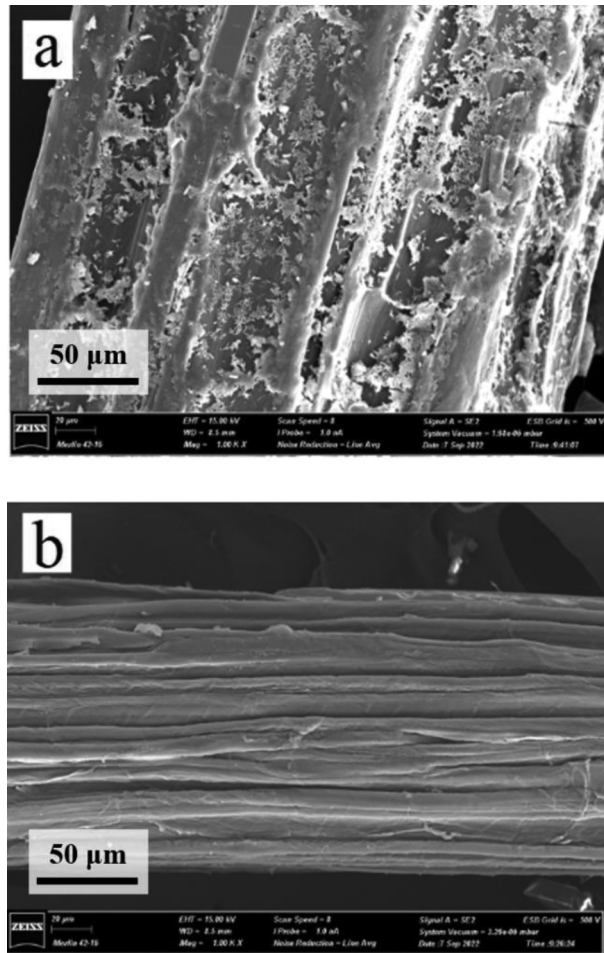


Figure 4. Schematic representation of the fiber-matrix interaction by interlocking after mercerization process.



**Figure 5.** SEM micrographs taken by a Carl Zeiss NTS at 15kV and magnification 1000x. (a) Untreated sisal fibers showing bright white regions all over the fibers' surface. (b) Sisal fibers treated with 2wt% NaOH for 2 h and rinsed with distilled water until pH is reduced back to 7.

degradation due to the resulting more ordered physical structure (Shesan et al. 2019; Xue, Tabil, and Panigrahi 2007). The alkali treatment has a lasting effect on the fiber mechanical properties such as strength and stiffness.

Sisal and hemp fibers were studied as a reinforcement in an epoxy matrix by Venkatesha Gupta, Akash, and Arun Kumar (2016), who treated the fibers with a 10% alkaline solution (NaOH) and developed a fiber-epoxy composite using the compression molding method (Venkatesha Gupta, Akash, and Arun Kumar 2016). The NaOH treatment was aimed to remove cementing materials present in the fiber and increasing the effective surface area of the fiber, therefore leading to a better fiber-matrix adhesion. Different percentages of fiber reinforcement, ranging from 10% to 50%, were used, and a comparative study of the resulting composites was made analyzing the tensile strength, flexural strength, and hardness. After the alkaline process, the composite containing 40% by weight of fiber reinforcement resulted in an enhancement value for tensile and flexural strength. This enhancement in mechanical properties was attributed to the possible chemical bonding at the fiber-matrix interface. The hardness properties were found to increase with increasing fiber content, due to the higher density of the reinforcement. Furthermore, the effect of the alkali treatment on the mechanical properties would depend on the alkali concentration, temperature and reaction time. According to

Rajkumar et al. (2018), the use of NaOH and isocyanate treatments to modify the sisal samples used as reinforcement in biobased epoxy resins improves the mechanical properties of the composite, and that is correlated to the increase in the surface energy of the treated fiber. It was shown through a quantitative theoretical approach that the modified fiber has higher surface energy compared to that of the resin and this thus yields higher tensile strength in the sisal bio-based epoxy composite, as it facilitates improved wetting between the materials during the fabrication of the composite (Rajkumar et al. 2018). Ashok Kumar et al. (2010) treated sisal/e-glass hybrid reinforcements with 5% NaOH soaked for 1 h. The fiber lengths were set to 1, 2 and 3 cm and embedded into an epoxy matrix. It is reported that the alkali treatment improved the hardness, impact strength, friction coefficient and chemical resistance. Furthermore, it is shown that the degree of improvement is a function of the fiber length where the 2 cm fibers obtained the best mechanical properties.

### **Acetylation methods**

Plasticization of natural fibers by the esterification process has also been used to reduce moisture absorption (Kalia et al. 2009). The introduction of functional groups by acetylation methods is an important route to reduce the hydrophilic organic behavior of the fiber, improving environmental degradation and dimensional stability. The substitution of cellulosic hydroxyl groups in the cell wall, mainly from the amorphous part, by the introduction of functional groups lead to this result (Kalia et al. 2009). Sukmawan et al. (2023) treated sisal fibers with acetic anhydride after pre-treating the fibers with alkaline hydrogen peroxide. Cellulose nanofibers were mechanically isolated using a high-speed blender, and it was reported that the acetylation process reduced the crystallinity index from 79% to 66% whilst the hydrophobicity was improved considerably. The sisal derived cellulose nanofibers were not used to produce composites.

The acetylation process on sisal fibers was carried out by Lopes et al. (2010) at various temperatures and for different durations. The effectiveness of the treatments, in terms of reducing hydrophilicity while preserving the mechanical properties, was assessed using water sorption, mechanical tensile tests, and infrared spectroscopy. Acetylation resulted in a significant reduction in water sorption, up to 50%, when compared to untreated fibers. Fiber treated for 3 h led to a decrease in mechanical properties, while treatment at 120°C for 1 h demonstrated improved physico-mechanical properties and an increase in hydrophobicity (Lopes et al. 2010).

### **Benzoylation and etherification**

Benzoylation and etherification of fibers are interesting methods to modify the hydrophilic nature of the fiber surface, thereby increasing the adherence with the hydrophobic polymer matrix (Kalia et al. 2009; Mishra et al. 2003). Firstly, it is necessary to treat the fiber with an alkaline reagent to produce a charge intermediate group, which reacts with groups such as epoxides, acrylonitriles, and benzyl chloride by nucleophilic addition (Kalia et al. 2009). Although no study was focused on benzoylation of sisal fibers embedded in epoxy, Sreekumar et al. (2008) studied the thermal behavior of 30 mm long sisal fibers treated with sodium hydroxide, benzoyl chloride and potassium permanganate separately embedded in polyester. It is reported that all coupons with treated fibers showed a reduction in the hydrophilic nature and lower water absorption. In particular, benzoylated-treated fibers showed the best thermal stability out of all the chemical treatments. Comparing the SEM of benzoylated fibers with that of raw fibers showed that the benzoylation treatment causes small voids and produces a rougher surface for the fiber which aids the resin impregnation process (Sreekumar et al. 2008). The surface modifications reported in (Sreekumar et al. 2008) agree with an earlier study conducted by Nair, Diwan, and Thomas (1996) where the benzoylated sisal fibers showed a reduction in tensile stiffness whilst the tensile strength and elongation at break remained relatively unchanged.

### **Coupling agents**

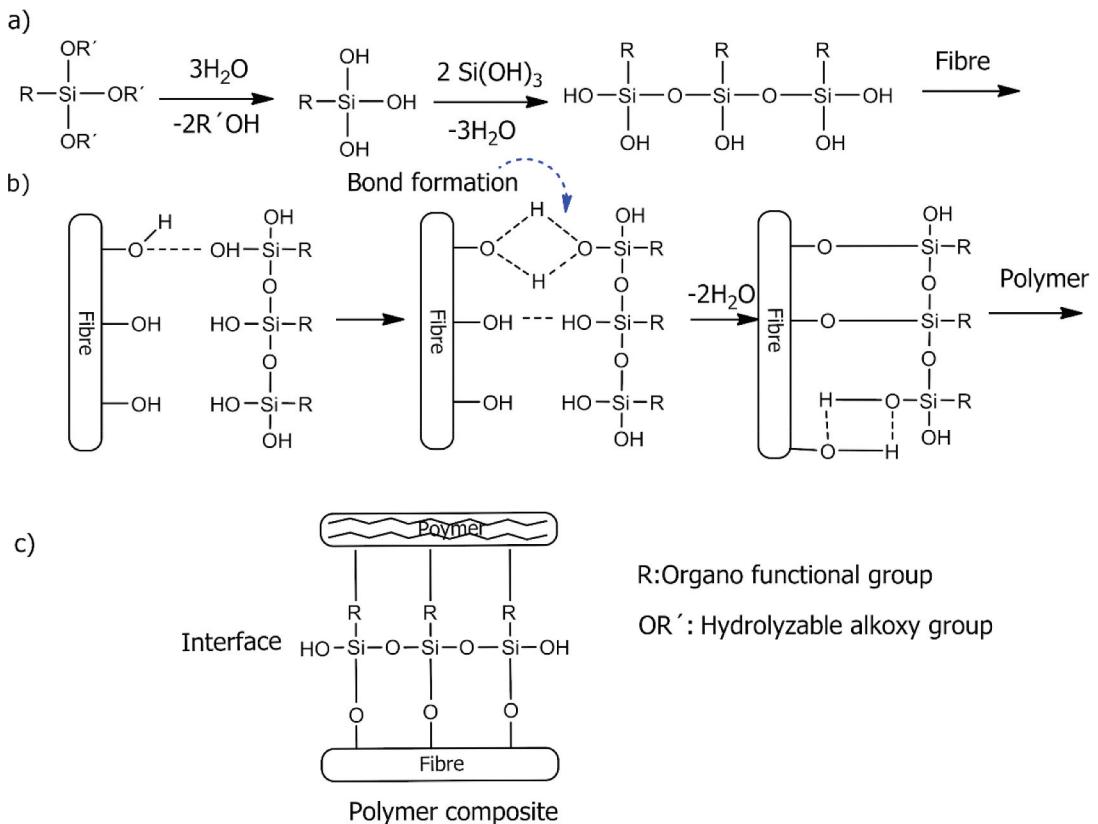
The use of coupling agents is a common method to improve the degree of crosslinking in the interface region of the composite, enhancing the adherence between the fiber and the matrix and increasing the

fiber wettability with the polymer matrix. As a result, the hydrophilicity of the fiber is reduced due to the substitution of the hydroxyl groups with the oxygen present in the fiber surface through chemical bonding (Ahmad et al. 2019).

An example of coupling materials is the use of silane as a coupling agent for natural fiber polymer composite. This can be represented schematically using the general formula of an alkoxysilane  $R-(CH_2)_n-Si(OR')^3$ , where “R” is an organofunctional group, which would react with the polymer to form a chemical bond (Figure 6). As can be seen, during hydrolysis, condensation of the alkoxysilane leads to silanols which become chemically linked with the cellulose fibers by the formation of covalent bonds. As a result, the number of hydroxyl groups on the wall surface is reduced, leading to decreased moisture pickup, while the surface of the fiber increases its hydrophobicity, which improves the moisture repellence of the final composite (Bledzki and Gassan 1999; Kalia et al. 2009; Pickering, Efendy, and Le 2016).

Gañan et al. (2005) highlighted that these silane treatments eliminate the cementing materials and hemicellulose compounds, which can be observed through FTIR analysis typically used to determinate the chemical functionalities of the material. The spectra showed that the carbonyl band located around  $1740\text{ cm}^{-1}$  almost disappears and there is a decrease in the intensity of the bands located around  $3300\text{ cm}^{-1}$  and  $1250\text{ cm}^{-1}$  which are related to the hydroxyl groups of carbohydrates and C-O ring of lignin, respectively (Gañan et al. 2005).

Rong et al. (2001), has evaluated different treatments including silane to modify the sisal structure used as reinforcement in epoxy resin. The reaction of the fiber with the chemical takes out the existing



**Figure 6.** Schematic reaction of (a) alkoxysilanes hydrolysis, (b) bond formation stage between a coupling silane agents and natural fiber, (c) fiber-polymer matrix bonding mechanism. R: representing organic group. Dash line: representing hydrogen bonding. OR' representing hydrolysable alkoxy group.

moisture while improving dimensional stability and enhances fiber dispersion in the polymer matrix. As a result, a significant improvement in the adhesion interface was observed, where the epoxy matrix penetrated the vacancies on the surface and inside the fiber bundles, obstructing the pull-out of the cells. The reduction of voids or pulled-out fibers in the composite surface after tensile strain test indicates an enhancement of the interface between the fiber and the resin (Fortea-Verdejo et al. 2017; Meenakshi and Krishnamoorthy 2019; Rong et al. 2001; Senthilkumar et al. 2018; Tragoonwichian, Yanumet, and Ishida 2007). The improvement in the compatibility due to silane treated fibers was also observed through SEM images reported by Rong et al. (2001), by identifying the surface morphology change due to the interfacial bonding adhesion of fiber-reinforced samples. Treatments such as silanization create stable covalent bonding between the chemical and the hydroxyl group of the fiber in the interface, and the cross-linked structure that is formed decreases the swelling of the composite. However, an added large coupling molecule reacts with the hydroxyl groups, which would destroy the cellulose packing chain leading to a decrease in the crystallinity and reducing the tensile strength whilst improving flexural performance (Rong et al. 2001).

Graft copolymerization is an effective methodology to improve the compatibility between the fiber and polymer matrix, which have different polarity. In this treatment, a functional group reacts with the chemical groups in the fiber, mainly with the active sites C2, C3 and C6 hydroxyls and C-H groups located in the cellulose structure, which get involved in an ionic or condensation polymerization (Agrawal et al. 2000; Kalia et al. 2009) (Figure 1). Dai and Fan (2014) observed that the organic material acts as a bridge between the hydrophilic reinforcement and the hydrophobic matrix by inducing the formation of covalent bonds and physical interaction. The binding occurs through a condensation reaction or the formation of hydrogen bonds in between both, the hydrophilic and hydrophobic parts (Dai and Fan 2014; Gañan et al. 2005; Gurunathan, Mohanty, and Sanjay 2015) (see Figure 1). Luo et al. (2016) managed to graft hyperbranched liquid crystal polymers with three-dimensional space structures onto microcrystalline cellulose fibers extracted from sisal fibers. The hyperbranched liquid crystals react with the functional groups located in the epoxy resins via covalent incorporation, resulting in an efficient interfacial fiber–epoxy interaction. When 1% of the hyperbranched liquid crystal was used as filler loading in the epoxy resin, the composite material showed an increased flexural strength, tensile strength, impact strength and flexural modulus by 60%, 69%, 130%, and 192%, respectively, due to the higher cross-linking densities in the composite and a stronger network structure (Luo et al. 2016).

### **Combined treatments**

Ahmad et al. (2019) suggested that the choice of chemical and physical treatments for fiber modification would depend on the required properties to be achieved. However, a combination of different methods could be used to optimize the composite, as emphasized by (Rong et al. 2001). Combining different methods can produce clean fibrils with high cellulose concentration from the fiber bundles, facilitating chemical reactions (Gañan et al. 2005). For instance, a pre-treatment of sisal fiber with alkali followed by a heating process has shown an increase in the flexural strength and stiffness in the fiber/epoxy composite. According to (Tragoonwichian, Yanumet, and Ishida 2007), the use of alkali treatment on sisal fiber followed by silanization could lead to a readily condensation reaction between the silanol groups of silane and hydroxyl groups (OH) of the fiber, enhancing the deposition efficiency (Tragoonwichian, Yanumet, and Ishida 2007). The result occurs because of the hydroxyl groups of cellulose fibrils becoming more exposed on the surface of the fiber and this increased the hydrophobicity of the material. In the research conducted by Gañan et al. (2005), an improvement in the adhesion between the fiber and epoxy matrix was observed using a combined method where the sisal fiber was modified with NaOH to remove the amorphous components, followed by a silane coupling agent treatment, introducing changes in the chemical structure and sisal properties, and enhancement of wettability of the material. The combination of both alkali and glutamic acid reinforced epoxy resin was investigated by Behera et al. (2022), where the chemical modification was evaluated using FTIR,

XRF and TGA. The study showed an improvement in the thermal stability, an increase in surface roughness and crystallinity and an enhancement in the wear resistance of the modified composite.

The hybridization of synthetic fiber and natural fiber has emerged as a viable solution to address the limitations of fiber-reinforced composites, as well as the environmental burden due to polluting materials. M. K. Gupta et al. (2015) conducted a study where sisal and glass fiber were mixed and used as reinforcement in an epoxy resin. This hybridization results in increased mechanical properties of the composite due to an improved adhesion interface, which minimized the voids present in the material. Tensile properties were analyzed to confirm the enhancements.

### Summary

A summary of chemical sisal fiber surface modifications is listed in Table 3. Note that most of the studies quoted in Table 3 include additional secondary processes like washing thoroughly with distilled water to balance the pH of the fibers and drying at certain temperatures and durations. For simplicity, these secondary processes are not listed in Table 3 hence the reader is encouraged to refer to the cited source for more details.

### Conclusion

Several techniques have been employed by researchers to improve the adhesion properties of sisal fibers. These adhesion properties play a crucial role in enhancing the global properties of fiber/resin composites. These techniques involve treating the fibers with chemical solutions while varying reaction parameters such as time, concentration, and temperature. Surface treatments of the sisal fiber have been used to alter the chemical structure of sisal fibers and introduce reactive groups on their surface, leading to significant changes in their physicochemical and mechanical properties. Techniques such as FTIR spectroscopy and SEM have proven invaluable tools in analyzing qualitatively the effect of fiber surface modifications. On the other hand, thermogravimetric analysis and mechanical testing, including tensile, flexural and impact, exposed the treatments' effect on the thermal stability and mechanical properties of the sisal fibers and their composites.

It should be noted that it is difficult to compare quantitatively the findings of different studies due to differences in procedures and parameters adopted, yet it is overall shown that fiber surface modification processes improve the adhesion between the sisal fiber and epoxy polymer matrix. The improvement in the interface is brought about by favorable wettability, mechanical interlocking, and better interaction through chemical bonding between fiber and resin. Chemical treatments have shown to be effective in increasing the compatibility between the sisal fiber and epoxy matrix by modifying the chemical structure of the fiber and increasing the contact area of the reinforcement and the polymer compound. The literature demonstrates that surface treatments of sisal fiber have shown different levels of success in improving fiber-epoxy resin compatibility. From the literature survey presented in this paper it is shown that the most popular chemical treatment is washing in NaOH solution followed by a rinsing process to reduce the pH back to neutral and then drying. In all NaOH treatment cases, the researchers observed cleaner fibers after the alkali treatment, and this promoted better adhesion with the epoxy matrix. Furthermore, silane treatments showed an improved resistance to fiber moisture uptake. In addition, it was observed that thermal treatments increase the crystallinity of the cellulose fibers thus modifying the morphology leading to stiffer composites. It was also observed that when the adhesion between the sisal fiber and epoxy matrix is improved, the impact strength is adversely affected because energy absorbing mechanisms, like fiber pull out, are hindered by the good adhesion between the two phases as pointed out by Kuruvilla et al. (1996).

Future efforts can be more focused on the interaction between sisal fibers and biobased epoxy resins, which helps in making these versatile composites even greener. In addition, future research should be motivated to find and utilize nontoxic, eco-friendly and economical chemicals to treat natural fibers to enhance the fiber-matrix compatibility. Furthermore, the production of sisal mats

**Table 3.** Summary of observations with respect to the sisal fiber chemical treatment. Note that a “+” signifies that the chemicals were added simultaneously, whereas a “→” implies the order of the treatments.

Fiber treatment	Composite	Composite fabrication	Notable observations	Ref.
5% NaOH	hybrid (sisal+eglass) - epoxy	Compression molding	<ul style="list-style-type: none"> <li>The alkali treatment improved the hardness, impact strength, friction coefficient and chemical resistance</li> <li>The degree of improvement is a function of the fiber length</li> </ul>	(Ashok Kumar et al. 2010)
5% NaOH	sisal - epoxy	Hand layup and compression molding	<ul style="list-style-type: none"> <li>Treated fibers composite showed an increase in the tensile and flexural strengths by 50% and 10% respectively.</li> </ul>	(Singh et al. 2017)
10% NaOH	hybrid (sisal+coir) - epoxy	Compression molding	<ul style="list-style-type: none"> <li>Most of the lignin, pectin and impurities were removed resulting in rougher fiber surface</li> <li>Small voids were observed on the fiber surface</li> </ul>	(Akash et al. 2016)
10% NaOH	hybrid (sisal+hemp) - epoxy	Compression molding	<ul style="list-style-type: none"> <li>Hybrid composite with 40wt% sisal/hemp fiber were found to possess the best tensile and flexural strengths</li> <li>Maximum hardness was observed at 50wt % which is the highest fiber to resin ratio tested.</li> </ul>	(Venkatesha Gupta, Akash, and Arun Kumar 2016)
<ul style="list-style-type: none"> <li>NaOH</li> <li>NaOH → silane</li> </ul>	hybrid(sisal+jute) - epoxy	Hand layup and compression molding	<ul style="list-style-type: none"> <li>Both chemical treatments improved the tensile properties</li> <li>The alkali treatment improved the flexural properties</li> <li>The mixed treatment decreased the flexural properties</li> <li>Impact strength was improved by the alkaline treatment</li> </ul>	(Cavalcanti et al. 2019)
<ul style="list-style-type: none"> <li>NaOH at 2%, 4% and 6% wt</li> <li>silane treatments</li> <li>NaOH → silane solutions</li> </ul>	sisal – benzoxazine + bisphenol A type epoxy	Compression molding	<ul style="list-style-type: none"> <li>The alkali treatment before silanization enhanced the efficiency of silane deposition on the fibers thus improving the fibers’ hydrophobicity</li> <li>Best fiber matrix adhesion was obtained with NaOH followed by silane treatment</li> <li>Decrease in impact strength for all fiber treatments</li> </ul>	(Tragoonwichian, Yanumet, and Ishida 2007)
<ul style="list-style-type: none"> <li>2% NaOH</li> <li>Isocyanate treatment</li> </ul>	sisal – 30% biobased epoxy	Vacuum infusion	<ul style="list-style-type: none"> <li>Treated fibers showed better adsorption/ wetting performance</li> <li>Treated fibers showed better tensile and flexural strengths</li> <li>Both fiber treatments had an adverse effect on the impact strength</li> <li>Both fiber treatments increased the thermal stability of the fibers</li> </ul>	(Rajkumar et al. 2018)
<ul style="list-style-type: none"> <li>2% wt. NaOH</li> <li>18% NaOH → 50% acetic acid</li> <li>cyanoethylated</li> <li>Silane coupling agent</li> <li>Heated air</li> <li>NaOH → silane coupling agent</li> <li>NaOH → Heat</li> </ul>	<ul style="list-style-type: none"> <li>sisal fibers alone</li> <li>sisal - epoxy</li> </ul>	Compression molding	<ul style="list-style-type: none"> <li>Chemical methods provide the fibers with higher extensibility through partial removal of lignin and hemicellulose and lower modulus</li> <li>Thermal treatment resulted in higher fiber stiffness and strength due to the increased crystallinity of hard cellulose.</li> <li>Cyanoethylated fibers showed reduced crystalline cellulose</li> <li>Silane treated fiber composites showed higher tensile and flexural strengths over a wide range of fiber volume fractions when compared with untreated fiber composites.</li> <li>Untreated fiber composites are stiffer than silane treated fiber composites over a wide range of fiber volume fractions</li> <li>Alkali and heat-treated fiber composites showed improved tensile properties.</li> </ul>	(Rong et al. 2001)

(Continued)

**Table 3.** (Continued).

Fiber treatment	Composite	Composite fabrication	Notable observations	Ref.
<ul style="list-style-type: none"> <li>• NaOH 20% wt.</li> <li>• Silane</li> <li>• NaOH → silane solution</li> </ul>	sisal - epoxy	Compression molding	<ul style="list-style-type: none"> <li>• Combining chemical and physical treatments has the potential to optimise natural fiber composites</li> <li>• Treated fibers showed better flexural properties</li> <li>• Best improvement in flexural strength was observed with NaOH → silane treatment</li> <li>• All fiber treatments increased the thermal stability</li> </ul>	(Gañan et al. 2005)
alkali (NaOH), glutamic acid	sisal - epoxy	Compression molding	<ul style="list-style-type: none"> <li>• Improvement in surface roughness, crystallinity, and thermal stability of chemically treated fibers in comparison to untreated fibers.</li> <li>• Microhardness properties of chemically treated sisal fiber showed minor improvement.</li> <li>• Increase in the dry wear resistance of chemically modified fibers relative to untreated.</li> <li>• Best wear properties were demonstrated by alkali treated fibers.</li> </ul>	(Behera et al. 2022)
<ul style="list-style-type: none"> <li>• dewaxing (benzene +alcohol at 1:1)</li> <li>• mercerisation (dewaxing → NaOH)</li> <li>• mercerisation → silane solution</li> </ul>	<ul style="list-style-type: none"> <li>• sisal fibers alone</li> <li>• sisal - epoxy</li> </ul>	Compression molding	<ul style="list-style-type: none"> <li>• Silane treated fibers and composites showed considerable resistance to moisture uptake</li> <li>• Water absorption of composites with mercerised fibers was lower than untreated fibers</li> <li>• Mercerisation and silane treatments improved the compressive strength</li> </ul>	(Bisanda and Ansell 1991)
hyperbranched liquid crystals grafted on silane treated sisal fibers	sisal microcrystalline fibers - epoxy	Not mentioned	<ul style="list-style-type: none"> <li>• The flexural, tensile and impact strengths were improved</li> </ul>	(Luo et al. 2016)
alkaline hydrogen peroxide + acetic anhydride	just nanofibers	N/A	<ul style="list-style-type: none"> <li>• Reduced the crystallinity index from 79% to 66%</li> <li>• Hydrophobicity was improved considerably</li> <li>• higher thermal stability</li> </ul>	(Sukmawan et al. 2023)
acetic anhydride and acetic acid at 3:2 by wt.	just fibers	N/A	<ul style="list-style-type: none"> <li>• Significant reduction in water sorption (up to a 50%), when compared to untreated fibers.</li> <li>• Treatment at 120°C for 1 hour demonstrated improved physico-mechanical properties and an increase in hydrophobicity</li> </ul>	(Lopes et al. 2010)

for industrial purposes shall be sought for to ease the manufacturing aspect of sisal composites. It was shown that compression molding is the prevalent way of manufacturing when it comes to sisal and epoxy composites. Bisanda (1993) produced economic roofing panels by compression molding using sisal fibers and naturally occurring phenol-based resin. It should be noted that although compression molding is a simple method of producing composites, it is usually limited when it comes to complex shapes and large dimensions like boat hulls. It would be interesting to focus future research toward studying the effect of fiber surface treatment when combined with manufacturing methods like resin transfer molding processes such as the widely used vacuum resin infusion.



## Highlights

- Fiber surface modification processes improve the adhesion between the sisal fiber and epoxy polymer matrix which is brought about by favorable wettability, mechanical interlocking, and better interaction through chemical bonding between fiber and resin.
- The most popular chemical treatment is washing in NaOH solution followed by a rinsing process to reduce the pH back to neutral and then drying.
- Silane treatments showed an improved resistance to fiber moisture uptake, whereas thermal treatments increase the crystallinity of the cellulose fibers, thus modifying the morphology leading to stiffer composites.
- When the adhesion between the sisal fiber and epoxy matrix is improved, the impact strength is adversely affected because energy absorbing mechanisms like fiber pull out are hindered.
- It was shown that compression molding is a prevalent way of manufacturing when it comes to sisal and epoxy composites and because of this production of sisal mats for industrial purposes shall be sought to ease the production aspect of sisal composites.

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## Disclosure statement

No potential conflict of interest was reported by the author(s).

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## Data availability statement

No new data were created or analyzed during this study. Data sharing is not applicable to this article.

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