

Sisal Fiber Reinforced Polymeric Composites: A Review**Adane Dagnaw Gudayu^{1,2}, Dr. Leif Steuernagel², Prof. Dr. Ing. Dieter Meiners², Dr. Rotich Gedion¹**¹Ethiopian Institute of Textile and Fashion Technology (EiTEX), Bahir Dar University, Bahir Dar, Ethiopia,²Institute of Polymer Materials and Plastics Engineering (PUK), Clausthal University of Technology, Clausthal-Zellerfeld, GermanyE-mail: habtabebaw@gmail.com, leif.steuernagel@tu-clausthal.de, Dieter.Meiners@tu-clausthal.de, rotichgedion2016@gmail.com

Abstract: Natural fibers are abundant, renewable, and environmentally friendly. As a result, they are being increasingly used as reinforcement with polymers and plastics to manufacture composite materials for both industrial applications and research. Sisal fiber, a natural fiber, is being progressively used in natural fiber composite applications. This paper reviews different kinds of literature and investigations made with sisal fiber reinforced polymeric composites, its properties, fiber modification techniques, and mechanical characteristics of its composites.

Key Words: Agave, Composite, Sisal, Polymer.

I. INTRODUCTION

Composites can be reinforced either by synthetic fibers or by natural fibers. Synthetic fibers-based composites are manufactured according to their constituents and their proportions, while the properties of natural fibers are unpredictable [1]. Recently, natural fibers have become popular due to their advantages over conventional reinforcements in terms of global warming, waste disposal and availability. As compared to other reinforcing fibers, natural fibers possess unique properties like low density, biodegradability, low-cost and nonabrasive.

In human history, natural fibers play a significant role in day-to-day life. For centuries, mankind has been solely dependent on plants and their fibers for their needs. The first uses of composites date back to the 1500s B.C., when early Egyptians and Mesopotamian settlers used a mixture of mud and straw to create strong and durable buildings, pottery and boats. Later, in 1200 AD, the Mongols invented the first composite bow for hunting and military application using a combination of wood, bone, and animal glue [2, 3]. Until the beginning of the 20th century, natural fibers and glues continued to be the only source of composite materials[2]. It was in the early 1900s that plastics were developed and in 1935, fiberglass was invented leading to the start of modern composite production [2, 3, 4]. Fiberglass and carbon fibers continued to be the major sources of reinforcement in the composite world. These minerals and oils are, however, scarce, non-renewable, non-biodegradable and environmentally unsafe. As a result, there is a need to use renewable, abundant, economical and biodegradable raw materials as an alternative or a substitute for the scarce minerals and petroleum byproducts. The recent composite production demands the use of natural fibers as reinforcement to polymeric matrices. The main reasons are the depletion of non-renewable resources such as petroleum byproducts and scarce minerals, cost-related factors [2,4], the resulting environmental problems during processing and disposal, and the consequent government regulations on waste management [2, 5].

Sisal is a leaf fiber that is part of the agave fibers family called *Agave sisalana* [6]. It is the main leaf fiber produced in the world, accounting for approximately 70% of the commercial production of all leaf fibers. In 2006, the worldwide production of sisal was estimated at 427,000 tons, according to the Food and Agriculture Organization Statistics published in 2006 [6]. Sisal plant produces about 200-250 leaves, and each leaf contains 1000-1200 fiber bundles composed of 4% fiber, 0.75% cuticle, 8% dry matter and 87.25% water [1, 2, 4]. Sisal is a lingo-cellulosic fiber. The primary chemical constituents, as studied by different researchers, is shown in Table 1.

Table 1: Main chemical constituents of sisal fiber

Cellulose (%)	Hemicellulose (%)	Lignin (%)	References
67-78	10-14	8-10	[1]
66-78		8-14	[6]
70		12	[7]

Sisal fibers are multicellular, with small individual cells bonded together. Single sisal fiber is a composite structure containing cellulose microfibrils which are reinforcements with lignin and hemicellulose as a matrix. Therefore, the cell wall is a composite structure of lignocellulosic material reinforced by helical microfibrillar bands of cellulose, as shown in Figure 1 [1,4,6,7].

II. SISAL FIBER AS A POLYMER REINFORCEMENT

Various research studies indicate that the mechanical properties of sisal fibers depend on cellulose content, the fibrillar angle and the lamellae matrix [1-7]. In comparison with other natural fibers, sisal fibers have salient features that make them essential for technical applications. Sisal has lower cellulose content with higher tensile and stiffness properties, maximum impact toughness, higher work of rupture and excellent acoustic absorption properties [4,6].

Despite the attractive features of natural fibers being renewable resources with minimum environmental impact, being biodegradable and biostable, their commercial use in the area of composites is still limited. This is because of their hydrophilic nature which makes them susceptible to moisture, temperature and ultraviolet light that cause degradation of the physical, mechanical and thermal properties of the resulting composite [1, 4, 8]. Jorg *et al.* [6] state that natural fibers have high water absorption and thermal stability problems.

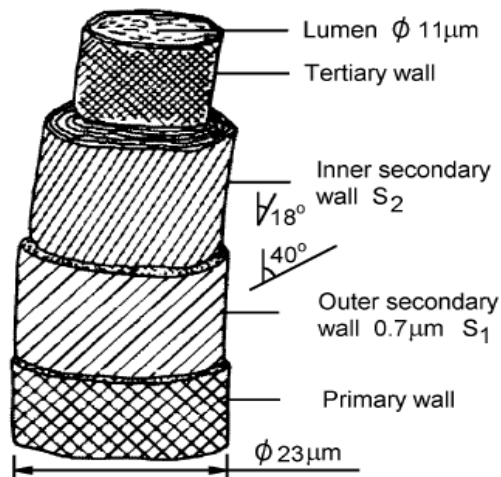


Figure 1: Cell wall structure of sisal fiber [7]

During application, the absorbed water leads to variable expansion behavior of fiber and matrix material, which, in turn, leads to the generation of interface stresses and cracks [1,6,8,9,10]. Besides, the matrix materials are often non-polar and hydrophobic, and chemical bonding to the polar and hydrophilic natural fibers is difficult [1,4,5]. For use in thermoplastic polymers, they are limited to those polymers with a lower melting point like polypropylene or polyethylene [10]. Thus, fiber surface modification techniques are used to minimize moisture absorption, increase thermal stability and promote compatibility between fiber and matrix. Oladele *et al.* [8] explained the importance of fiber/matrix interface to the micromechanical behavior of sisal fiber reinforced polymeric composites. Accordingly, one difficulty that has prevented the use of natural fibers is the lack of excellent adhesion with the polymeric matrix due to the incompatibility between the hydrophilic fiber and hydrophobic matrix, which leads to premature ageing by degradation and loss of strength [1,4,6,8,9]. Hao *et al.* [9] also discussed the dependence of mechanical properties of sisal fiber-reinforced polymer composites on the fiber/matrix interfacial strength in addition to the intrinsic properties of fibers and polymer matrix. The stress is transferred from the polymer matrix to the fibers via the interface of the composite under load-bearing. Therefore, a fundamental study of the fiber–matrix interface is critical to the development of fiber-reinforced polymer composites. Numerous studies have proved that a good interfacial adhesion is of great importance for obtaining good mechanical properties of composites materials [8, 9]. Several remedial procedures are tried to overcome these challenges; among them is chemical surface treatments. Many works confirm that chemical treatments of natural fiber are an effective way to improve the interfacial compatibility and adhesion of natural fibers with the non-polar matrices [6-29].

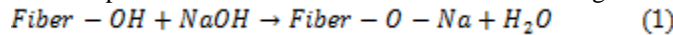
A. Sisal Fiber Modification Techniques

Hantao *et al.* [11] studied mercerization, acetylation, silane and permanganate treatments of sisal fiber. They concluded the importance of the procedures for facilitating excellent adhesion between sisal fibers and the PLA matrix. According to Hantao *et al.* [11], higher mechanical properties were obtained from the treated-sisal fibers/PLA bio-composites than pure sisal fiber /PLA bio-composite. Chianelli *et al.* [12] stated that adding untreated sisal fibers to recycled HDPE matrix contribute to a decrease in the strain due to a poor fiber/matrix interfacial adhesion. Similarly, C. Romão *et al.* [13] found an inferior result for the tensile strength and tensile

modulus of the untreated sisal composite than the treated equivalent. They explained that the result is due to inadequate fiber/matrix adhesion than the treated sisal fibers. The basic principle of fiber surface modification is the removal of impurities like wax, lignin, pectin and other extractives to make the fiber surface rough and thus compatible with the hydrophobic matrix. Some of the modification techniques make a change in the chemical groups on the structure of cellulose which result in chemical bonding with the hydrophobic matrix [6,9-11]. Some of the chemical surface modification techniques employed on sisal and other natural fibers are presented.

i. Alkali Treatment

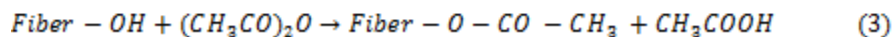
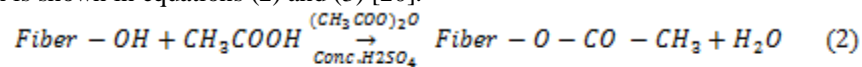
Alkali treatment of sisal involves immersing the fiber in a specific concentration of NaOH or other alkali solution for a specified period at room temperature or at elevated temperature [9, 10, 11, 14, 15, 16, 17]. The expected result in alkali treatment is the removal of some of the hydrogen bonding in the network structure and thereby increasing the amorphous content. The reaction mechanism during alkali treatment is shown in equation 1.



As explained by Bisanda [17], mercerization improves resin uptake, removes cuticle layers and waxy substances, and promotes chemical bonding. According to Bisanda, the process also has the effect of roughening the fiber surface through the dissolution of intra-crystalline and inter-crystalline lignin. When a wet matrix is brought in contact with the mercerized fiber, there is some capillary effect, that is, increased surface tension. Some form of mechanical interlocking between the rough fiber surface and the matrix is mostly responsible for the increased mechanical strength when fibers are mercerized. Since more resin is attracted to the mercerized fibers, the composite exhibits higher density [16, 17]. Bisanda [17] also demonstrated the interfacial strength of the resulting composite. Thus, in comparison with the untreated sisal/epoxy composite, the compressive strength of the mercerized sisal/epoxy composite is favorably improved as a result of a good fiber-matrix interfacial bond strength. The compression test is, therefore, suitable for assessing interfacial bonding characteristics in a composite system. During compression in the fiber direction, the interface is subjected to shear as the fiber tries to resist micro-buckling against the more rigid matrix. During compression, the fiber has to be supported against micro-buckling by a continuous coherent matrix. The presence of voids at the interface and poor alignment can lead to easy micro-buckling and therefore, initiation of compression failure. Based on the test, the compression strength of the alkali-treated sisal composite is higher than the untreated composite [16]. According to the pullout test, Mokaloba *et al.* [16] observed that both the acetylated, discussed in the subsequent section, and alkali-treated fibers exhibited improved interfacial shear strength compared to untreated samples.

ii. Acetylation

Acetylation is one of the most studied modification methods of cellulosic fibers for increasing the hydrophobicity of natural fibers. The main objective behind acetylation of sisal and other natural fibers is to react with hydroxyl groups of the fiber to change their hydrophilic properties to hydrophobic [10-11, 14-16, 19]. Mohebbi *et al.* [19] explained that the fibers react with the acetic anhydride and that the substitution of the hydroxyl groups (OH) of cellulose by the acetyl groups (CH₃CO) occurs over time rendering the fiber surface more hydrophobic. Acetylation is beneficial in reducing moisture absorption of natural fibers. A reduction of about 50% of moisture uptake for acetylated jute fibers and of up to 65% for acetylated pine fibers has been reported in the literature [18]. The reaction mechanism during acetylation is shown in equations (2) and (3) [20].



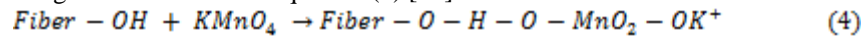
Acetylation of natural fibers with a catalyst (Eq. 2) and without catalyst (Eq. 3) [20, 24]

Kabir [20] studied the effect of acetylation treatment on fiber-matrix interaction; the modification makes the fiber surface rough giving a better mechanical interlocking with the matrix. The procedure also improves the fibers' dispersion in the matrix, thus enhancing the dimensional stability of the composite [20]. Fibers were acetylated with and without an acid catalyst to graft the acetyl groups onto the cellulose surface. In general, acetic acid does not react sufficiently with the fibers. As a result, it is necessary to use a catalyst to speed up the acetylation process. Acetic anhydrides, pyridine, sulphuric acid, potassium and sodium acetate are commonly used catalysts to speed up the acetylation process. However, strong acid catalysts cause hydrolysis of cellulose which results in damage to the fiber structure [20]. For this, the selection of catalyst is an essential factor for acetylation treatment. The acetylation process is also influenced by the reaction time. Longer reaction time allows acetic anhydride (catalyst) to access

fiber constituents. The reagent then reacts with hemicellulose and lignin constituents, removes them from the fiber, resulting in the opening of cellulose surface to allow reaction with the matrix molecules. Mwaikambo and Ansell [21] used acetic acid and acetic anhydride (catalyst) to treat hemp, flax, jute and kapok fibers. Cruz *et al.* [22] also investigated acetic anhydride treatment on different types of natural fibers and their effects on equilibrium moisture content. They reported improved moisture resistance properties of the treated fibers. This was due to the removal of active hydrogen groups in cellulose and hemicellulose constituents from the treated fiber. Mishra *et al.* [23] used acetic anhydride treatment, with glacial acetic acid and sulphuric acid, on an alkali pre-treated (5% and 10% NaOH solution for an hour at 30°C), sisal fiber and reported improved fiber-matrix adhesion of the final composites.

iii. Permanganate (KMnO₄) Treatment

Permanganate treatment of natural fibers is conducted with potassium permanganate (KMnO₄) in acetone solution [18, 20, 23]. This treatment forms highly reactive permanganate ions (Mn³⁺) which react with the cellulose (hydroxyl groups) and form cellulose-manganate and initiates graft copolymerization [12, 18, 20]. Kabir [20] further explained that this treatment enhances chemical interlocking at the interface and provides better fiber adhesion with the matrix. The therapy also reacts with the OH groups in lignin and removes them from the fiber; thus, the hydrophilic nature of the fiber is reduced. Higher concentrations of KMnO₄ (more than 1%) cause excess delignification from the fiber structure and degrade its strengths [24,11]. The reaction between the fiber-OH group and potassium permanganate are shown in equation (4) [20].



Potassium permanganate treatment of natural fibers [20, 24]

Kabir [20] conducted an oxidation reaction using KMnO₄ etching the fiber surface and making it physically rougher to improve mechanical interlocking with the matrix. Flexural strength and modulus properties were increased by 5% and 10% for the treated banana fiber reinforced in polypropylene composites. Li *et al.* [24] applied 0.2% KMnO₄ solution (in 2% acetone) on alkali pre-treated (2% NaOH for an hour) flax fiber. They reported treated fiber-LLDPE, and HDPE composites had higher tensile strength compared to the untreated samples.

iv. Silane Treatment

Silane is a multifunctional molecule used as an efficient coupling agent to modify fiber surfaces [12, 15, 16, and 20]. It is an inorganic compound with chemical formula SH₄. Silane coupling agents are known as efficient coupling agents widely used in composites and adhesive formulations. This type of agents is used as adhesion promoters or substrate primers, which offer strong adhesion. Silane molecules form a chemical link between the fiber surface and the matrix through a siloxane bridge. Silane undergoes several stages of hydrolysis, condensation and bond formation during the treatment process of the fiber. Silane forms silanol in the presence of fiber moisture [15, 20]. During the condensation process, one end of silanol reacts with the cellulose hydroxyl group and the other purpose reacts (bond formation) with the matrix (Sisal-Matrix) functional group. This co-reactivity provides molecular continuity across the interface of the composite. It also provides the hydrocarbon chain that restrains the fiber swelling into the matrix. As a result, fiber-matrix adhesion improves and stabilizes the composite properties [24]. Natural fibers exhibit surface micro-pores and silane couplings act as surface coatings to penetrate the pores. In this case, silane coating is used as a mechanical interlocking material for the fiber surface [20]. Cantero *et al.* [8] studied the effects of various fiber treatments on wettability and mechanical properties of the flax/PP composites. The maleic anhydride (MA), maleic anhydride-polypropylene copolymer (MAPP) and vinyl trimethoxy silane (VTMO) were used as coupling agents in this research. They observed that the composites with MAPP treated fibers achieved the highest mechanical properties. Meanwhile, there was no significant change in the value of composites containing the MA and VTMO treated fibers compared to the untreated one [8, 16].

III. MECHANICAL PROPERTIES OF SISAL FIBER REINFORCED POLYMERIC COMPOSITES

One of the main concerns of researchers in the area of material sciences and engineering is the mechanical properties of sisal reinforced composites. From the performance point of view, the material must fit the intended use or purpose. Therefore, mechanical properties are one of the most important parameters which are used to evaluate the material or product performance. As far as composites are concerned, tensile strength, modulus, flexural strength and impact strength are the significant mechanical properties used as a measure of performance [11, 25, 26, 27, 29].

A. Interfacial Adhesion Assessment

As discussed earlier, an excellent fiber/matrix interface is vital for improved mechanical properties. Mokaloba *et al.* [16] studied the effect of mercerization and acetylation on fiber-matrix adhesion. From the fiber pullout test result

shown in Table 2, Mokaloba *et al.* [16] concluded the following; first, chemical treatment strongly influenced fiber-matrix bond. The interfacial adhesions shear strength of the mercerized and acetylated fibers increased by 173% and 435%, respectively. Second, from the surface morphology by SEM analysis, mercerization and acetylation chemical modification treatments enhanced cellulose exposure which facilitates improved fiber-matrix interlocking. Based on these effects and improvement in interfacial adhesion characteristics, the employed two treatment methods are considered suitable for sisal fiber [16].

Table 2: Average values of recorded and calculated parameters from the fiber pullout test [16]

Composite	Debonding force (N)	Sliding friction (N)	Interfacial shear strength (Mpa)	%IFSS improvement
Untreated fibers	3.51	<1	0.48	-
Mercerized	9.56	3	1.31	173
Acetylated	18.3	5	2.57	435

v. Tensile Strength and Modulus

Tensile strength is the maximum stretching force that a material can resist before breaking or tearing apart [5]. It is a property used to assess the material's resistance towards tensile force. Tensile strength test of the composite is conducted as per ASTM D638 test standard [20, 21]. M. Kumar *et al.* [26], found that the tensile strength and tensile modulus of sisal/epoxy composites increased with increasing sisal fiber content up to 30 wt.%. Accordingly, tensile strength and tensile modulus of the composite at 30% sisal were found to be 83.96 MPa and 1.580 GPa respectively, which are 148% and 122% more than that of pure epoxy [20]. Zou *et al.* [9] compared neat polylactic acid (PLA) with sisal/PLA composite. They found that the tensile strength and Young's modulus of the untreated-SF reinforced biocomposite increased by 29.3% and 57.6%, respectively. The increase in modulus is due to the introduction of higher modulus sisal fibers into the PLA matrix, which indicates the occurrence of efficient stress transfer from the PLA matrix to stiffer sisal fibers [9].

Furthermore, among the different sisal fiber surface treatments, mercerization is proved to increase tensile properties of sisal composites [11, 8, 12, 15, 116, 17, 19, 28]. A review by Kumar *et al.* [14] reported the mechanical behavior of sisal fiber and epoxy resin matrix composite at 30%, 35% and 45% fiber loading. Sisal fiber was treated in NaOH aqueous solution for 1 hour at room temperature. The maximum tensile strength of 30MPa was obtained for treated fiber at 45% fiber loading, which is approximately three times higher than 9.25 MPa of the untreated fiber due to the surface modification which promoted good wettability and better fiber-matrix adhesion, allowing efficient stress transfer between the matrix and the fibers [14].

vi. Flexural Strength and Modulus

Flexural strength is the ability of a material to resist deformation under a bending load, and it is measured in terms of stress. It represents the highest stress experienced by the material at the rupture load [11]. Flexural properties of the composite are determined using a three-point bending test based on ASTM D790 test standard [10, 26, 28]. Flexural strength and flexural modulus were calculated using the following equations (5) and (6) [10].

$$\text{Flexural strength} = \frac{3FL}{3bd^2} \quad (5)$$

$$\text{Flexural modulus} = \frac{mL^3}{4bd^3} \quad (6)$$

Where F is the ultimate failure load (N); L , the span length (mm); b and d , the width and thickness of specimen in (mm) respectively; and m , the slope of the tangent to the initial line portion of the load-displacement curve [10].

Piedad *et al.* [19] investigated the introduction of untreated sisal fibers leading to significant improvement on flexural strength and modulus compared to the neat epoxy matrix. Thus, by loading a 40% wt. of untreated fiber, the flexural strength increases 36% with respect to neat epoxy. The variation of flexural modulus with fiber content follows a linear trend. These results are mainly related to the mechanical properties of sisal fibers. Kumar *et al.* [26] investigated the mechanical properties of sisal-epoxy composites. They discovered that the flexural strength and flexural modulus of sisal composite are also found to increase with increasing sisal fiber content up to 30 wt.%. The composite with 30% sisal offers the maximum value of flexural strength and flexural modulus due to strong fiber-matrix adhesion [20]. Kabir [20] showed that alkali treatment with concentrations of 6-10% NaOH yield improved flexural properties. This was because higher NaOH concentrations effectively removed the hemicellulose and lignin coverings from the fiber surface. As a result, more accessible cellulose surfaces were exposed for matrix adhesion

and thus produced strong interface bonding [28, 29]. Bledzki *et al.* [28] studied the effect of acetylation treatment on flax fiber. They reported that acetylated flax fiber/PP composites showed 25% higher tensile and flexural properties as compared to the untreated fiber composites. Thus, the flexural strengths were found to increase up to 18% degree of acetylation after which it was decreased rapidly with increasing degree of acetylation. The flexural modulus was also found to increase up to 18% acetylation slightly and fell slowly with increasing degree of acetylation.

vii. Impact Strength

The impact strength of composite material is its ability to absorb and dissipate energy in the form of creating new surfaces under shock or a sudden blow. There are two frequently used testing methods for determining impact strength - Charpy test and Izod test [11]. The Charpy impact test is a standardized high strain-rate test which determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of a given material's toughness. It acts as a tool to study the brittle-ductile transition [27]. Manoj *et al.* [26] used the Izod impact method based on ASTM D 256 test standard; Bledzki *et al.* [28] used the ASTM D 6110-04 test standard, Charpy impact method. As evident from Manoj *et al.* [26], impact properties also follow the same trend as tensile and flexural properties. They are found to increase with increasing sisal fiber content up to 30 wt. % in the epoxy matrix. Impact strength and impact energy are found to be maximum for composite with 30% sisal, 22.03 kJ/m² and 1.0909 J respectively, which are much higher than those of pure epoxy. Based on the conclusion by Zou *et al.* [9], surface treatments have a substantial effect on the impact property of the bio-composites, and the results are different for dissimilar treatment methods. The bio-composites with surface treatments have higher impact strength than the neat PLA matrix and the bio-composite. Therefore, mercerization is proven to increase the impact strength of sisal composites. Hantao Zou *et al.* found that the impact strength (3.7 kJ/m²) of mercerized sisal/PLA bio-composite improves by 16% over the untreated sisal/PLA bio-composite and 68% over neat PLA matrix, respectively [9]. As concluded by Bledzki, *et al.* [28], acetylation also increases the Charpy impact strength of flax fiber reinforced polypropylene composite, which even starts to decrease as the degree of acetylation increases beyond 18%.

IV. CONCLUSION

Many leaf fibers have attracted the interest of researchers. Among them is sisal. Sisal is a leaf fiber with high tenacity and tensile strength, abrasion resistance, alkali resistance, renewable, eco-friendly and is abundantly available. Despite its advantages, sisal, as a natural fiber, has high moisture absorption, which results in lower interfacial adhesion with polymer matrices and makes its application in the composite industry limited. Besides, it has relatively gentle mechanical properties than glass and carbon fibers. As explained in this review, physical and chemical surface treatments are very important for minimizing the problems. It can be generally concluded that fiber surface treatments increase the mechanical properties of sisal fiber reinforced polymeric composites.

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