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Study of the Effects of Alkali Treatment and Fiber Orientation on Mechanical Properties of Enset/Sisal Polymer Hybrid Composite

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Abstract: In the manufacturing process of innovative fiber-based composite materials, natural fibers are among the most commonly employed reinforcements. In this study, Enset/Sisal (E/S) fiber with a polyester matrix was used to develop the hybrid composites. Hand layup methods were employed for the sample preparation from untreated, 5%, and 10% alkali-treated unidirectional and woven fiber orientations having 50:50 volume ratios. The mechanical properties and water absorption of natural fiber hybrid composites were influenced by fiber treatment and orientation. In the present investigation, the result shows that treated and woven fiber orientation hybrid composites exhibit better mechanical properties than untreated and unidirectional E/S hybrid composites. The 5% NaOH-treated samples have higher tensile and flexural strength properties than the untreated and 10% alkali-treated composites, while the 5% NaOH-treated fiber composites have lower water absorption properties. The tensile and flexural strengths and impacts of 5% NaOH-treated composites were improved by 5.21%, 9.25%, and 5.98%, respectively, over untreated E/S hybrid composites. The morphological properties of the fracture surface of the composite were observed using scanning electron microscopy (SEM).

Keywords: hybrid composite; alkali treatment; mechanical properties; water absorption; scanning electron microscopy

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1. Introduction

In the modern industries of today, natural fiber-based polymer composites have potential applications to replace synthetic fiber-based polymer composites. This is due to the extensive availability of natural fibers in many areas, their low cost, biodegradability, light weight, and low energy consumption [1,2]. The most realistic industrial applications might include automotive, construction, aerospace, packaging, furniture, shipping pallets, electronics, medical, sports, etc. [3]. Although natural fiber-based polymer composites are now potential candidates to replace synthetic fiber-based composite materials that aid in pollution control, their mechanical performance is insufficient [4]. The mechanical performance of a composite material generally depends on the fiber types and matrix, fiber loading, fiber/matrix adhesion, fiber treatment, fiber length, fiber orientation, and hybridization [5,6]. Hybridizing is seen as a promising strategy to improve the mechanical properties of composites. The properties of single-fiber composites were improved by hybridizing with other fibers, which can mitigate the disadvantage of one fiber by others while keeping the advantages of others [2,7]. When two natural fibers are hybridized, the attention is often focused on getting a better balance in mechanical, chemical, and physical properties rather than optimizing the hybrid effect [8–10].

The treatment and orientation of the fibers, in addition to fiber hybridization, are the primary factors affecting how well natural fiber polymer composites function mechanically [11]. Natural fibers' hydrophilic nature causes them to interact poorly with hydrophobic polymeric materials, which restricts the ability of the composite parts to transfer stress. It is well known that composites' mechanical properties are impacted by the matrix's and fibers' poor adherence. In order to increase the interfacial adhesion and, consequently, the overall properties of the composite product, modifications have been a prominent issue in natural fiber-reinforced composites [8]. Chemical fiber treatments are the most popular and effective approach to cleaning the fibers of contaminants. The most popular chemical method for treating fibers is the alkali (mercerization) treatment, which removes weak components like lignin and hemicelluloses, wax oils, etc. from the cellulosic molecular structure to improve the roughness of the fiber surface. As a result, the natural fibers and the polymer matrix strongly interlock [12,13].

The molecules' alkali-sensitive hydroxyl (OH) groups are disassembled, which causes water molecules (H-OH) to interact and remove impurities from the fiber structure. According to the relationship below, the remaining reactive molecules create fiber-cell-O-Na groups in between the cellulose molecular chains.

$$\text{Fiber-cell} - \text{OH} + \text{NaOH} \text{ - - - - - - -> } \text{Fiber cell} - \text{O-Na}^{+} + \text{H}_2\text{O} + \text{impurities}$$

As a result, hydrophilic hydroxyl groups are diminished, and fiber moisture resistance properties are improved. Additionally, this improves the length-to-diameter aspect ratio by reducing fiber diameter and cleaning the fiber surface. The principal changes brought about by alkali treatment are an improvement in the fiber and matrix interfacial contacts and the elimination of hydrogen bonding in the network structure [14,15]. Analyzing the effects of NaOH on the mechanical and water absorption of an almond shell-sugarcane leaf hybrid composite, it was discovered that the treated fiber composite outperformed the untreated one [16]. Tensile and flexural properties of hybrid composites made of unsaturated polyester matrices with alkali-treated Palmyra Palm Leaf Stalk Fiber (PPLSF) and jute fibers were investigated, and both mechanical properties were improved. However, the impact strength of pure PPLSF polyester composites is greater than that of hybrid and pure jute polyester composites, demonstrating the shortcomings of single fiber composites that were overcome by fiber treatments and fiber hybridization [17]. The jute/kenaf/e-glass woven hybrid composite was studied, and the hybridization effects improved the impact and inter-laminar shear strengths. In addition, fiber hybridization improves composite debonding and microcracks [18]. The water absorption and surface hydrophobicity of kenaf/sisal hybrid composite were studied, and the result showed that higher water absorption and hydrophobicity were observed in kenaf/sisal hybrid composite when compared with a neat bioepoxy composite, due to the increased surface roughness of the composites [19].

The orientation of the fiber is the other key element that significantly influences the mechanical characteristics of fiber-based composites. The fiber orientations of a fiber-based composite was used to define its structure and attributes [2]. The physical properties of fiber composites are more influenced by fiber orientation than by fiber volume ratio [20]. The acoustic and mechanical properties of snake grass/waste tea leaf fibers (SGF/WTLF) with glass fiber (GF) were investigated, and the mechanical properties improved as the SGF in the hybrid composite was increased [21]. According to the research mentioned above, fiber treatment and fiber orientation are the key factors that influence the mechanical properties of hybrid composites made from natural fibers.

In the present study, untreated and NaOH-treated (5% and 10%) enset and sisal hybrid composites were fabricated in unidirectional and woven types. The effects of treatment and orientation on the mechanical and water absorption properties of the enset-sisal hybrid-reinforced polyester composites were analyzed. The morphological properties of the fracture surface of the composite were observed using scanning electron microscopy (SEM).

2. Experimental Materials and Methods

2.1. Materials

The enset fiber, extracted from psuedostem parts of the enset plant, and the sisal fiber, extracted from leaf parts of the sisal plant for this study, were collected from south-east Oromia, Ethiopia, due to the availability of fiber resources. Unsaturated polyester resin with the brand name "TOPAZ-1110 Phthalic Anhydride" was used, which was formed by condensation polymer reaction between a glycol and an unsaturated dibasic acid having a long chain with a number of carbon double bonds and has effective strength, good toughness, and resistance to moisture [22]. Wax was used to prevent the mold's surface from sticking to the composite. This unsaturated polyester, wax, and hardeners were bought from the World Fiber Glass Company and the Ethio-Plastic Company in Addis Abeba, Ethiopia. The Universal Test Machine (UTM), Charpy Impact Test Machine, and Scanning Electron Microscopy (SEM) were used to evaluate the mechanical and morphological behavior of the composites. Six different kinds of samples were made with a 30:70 fiber-to-matrix weight ratio and 50:50 ratios of the enset and sisal fibers. Two cases of fiber orientation, namely unidirectional and woven, were considered. In both cases, three different types of samples, i.e., (1) untreated, (2) 5% NaOH-treated, and (3) 10% NaOH-treated composites, were prepared and tested.

2.2. Methods

2.2.1. Fiber Treatment

The fibers were extracted from fully-fledged enset pseudostem and sisal leaf parts. The extracted fibers were washed and dried in sunlight for 8 h. Enset and sisal fibers were immersed in 5% and 10% NaOH (alkali) solutions for 2 h at room temperature. The immersed fibers were removed and washed several times with water until any NaOH that had adhered to the fiber surface, and the fibers were dried at room temperature for 24 h as recommended in [23].

2.2.2. Composite Fabrications

Fibers from both enset and sisal were extracted, dried, washed, and alkali-treated. The composites were prepared using the hand lay-up method. The sample was fabricated in the following dimension (300 × 300 × 5) mm on steel sheet plate. The composite samples were prepared at room temperature for untreated, 5%, and 10% NaOH with woven and unidirectional orientations, as shown in Figure 1a,b. The resin and hardener, in the ratio 10:1, were prepared and poured onto the mold surface. A roller was used to distribute the resin and again spread polyester and fiber, and this was repeated until the required lamina existed [24]. Then, the samples were compressed with a 30 kg load for 24 h, to ensure that the polyester resin had penetrated the porosity of the fibers and samples. To ensure matrix and reinforcement strength, the sample was cured for 48 h. After the curing process, test samples were cut to the required standards.

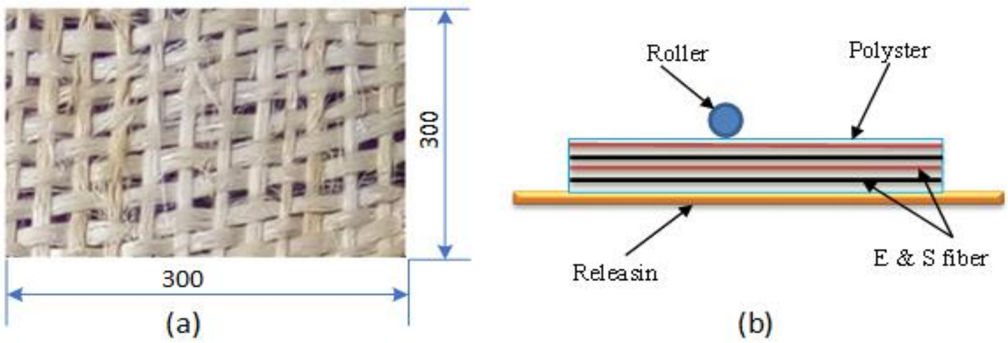

Figure 1. (**a**) A single layer of woven fibers; and (**b**) A molded sketch map.

2.2.3. Mechanical Properties

Among the many factors that affect the mechanical properties of the natural fibers, composite fiber treatment and fiber orientations are taken as the major factors in this investigation. Tensile testing is used to measure the force required to break the test specimen and the extent to which the specimen stretches or elongates to the breaking point. The prepared dimensions of the specimen were (250 × 25 × 5) mm, i.e., length, width, and thickness, respectively, as per ASTM D3039 standards [25]. A universal test apparatus (Bairoe, Shanghai, China, universal test apparatus) with a 50 kN tensile test capacity and test speeds of 5 mm/min crosshead speed and 150 mm gauge length was used for the experiment. Three specimens were tested for each set of samples, and an average value was taken. The specimens for the tensile test of untreated, 5% treated, and 10% NaOH treated unidirectional and woven are given in Figure 2a–c, respectively.

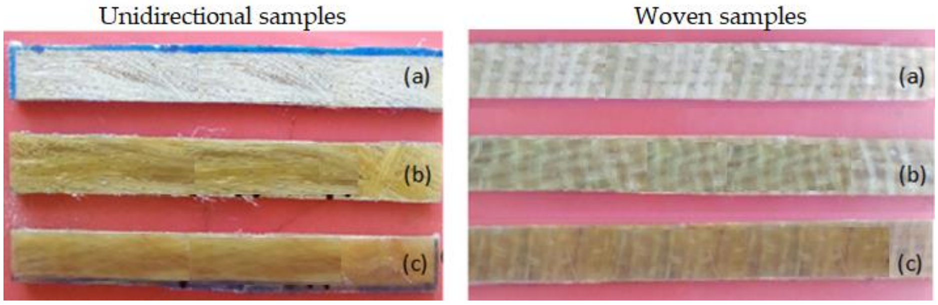

Figure 2. Unidirectional (L) and woven orientation (R) types of (**a**) untreated; (**b**) 5% NaOH-treated; and (**c**) 10% NaOH-treated E/S hybrid composites.

Flexural strength testing was done to determine the material's capacity to withstand deformation caused by a load. Rectangular cross-sections were used because they were either directly formed or cut from molded pieces. The three-point bend test is designed to encourage interlaminar shear failure. This test was conducted as per the ASTM D7264 standard for UTM testing [26] (WP 310 universal material tester, gunt, German). The test speed was maintained between 0.5 and 1 mm/min. The specimen dimension is (127 × 13 × 5) mm. The specimen for the flexural test is given in Figure 3. The specimen for the flexural test is given in Figure 3. The calculation of flexural strength and the flexural module was performed using the following formula: Equation (1) [25].

$$\sigma_f = \frac{3 \times p_{max} \times L}{2 \times b \times h^2} \quad (1)$$

where p_{max} = load maximum at failure, b = width of specimen, h = thickness of specimen, and L = length of specimen between the two support points.

(**a**) (**b**)

Figure 3. (**a**) Bending test setup in the UTS machine and (**b**) flexural test specimens.

The impact is a single-point test that gauges a material's resistance to an impact load from a swinging pendulum and keeps fracturing the specimen until it breaks. The dimension of the specimen used for the impact test with a V-notch was (65 × 13 × 5) mm, which was determined using a Charpy impact tester as per ASTM D256 [17] (Ceast Torino, Italy).

Water absorption properties of enset/sisal hybrid composites immersed in water at room temperature were studied as per ASTM 570 [27]. To study the water uptake, the specimens were submerged in normal water (pH = 7) at room temperature. The samples were taken out periodically and weighed immediately after cleaning the surface of a sample with a dry cloth and using a precise 4-digit balance to find out the content of water absorbed. Prior to being submerged once more in the water, all samples were dried in an oven until a constant weight was achieved. Using Equation (2), the amount of water absorption was calculated.

$$WA = \frac{m_2 - m_1}{m_2} \tag{2}$$

where WA = water absorption, m_1 and m_2 the weight of a dry and wet sample.

2.3. Scanning Electron Microscopy (SEM)

SEM is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The morphological characterization of the composite surface is observed in a scanning electron microscope of type M (Model JEOL, Kyoto, Japan). The composite samples were thoroughly cleaned, air dried, and coated with 100 A° of thick platinum in a JEOL sputter ion coater before being examined under SEM at 10 KV. The fracture surface morphology of the composite specimens is observed with SEM.

3. Discussion of Results

3.1. Tensile Strength Test

As shown in Figure 4, a 5% NaOH-treated fiber hybrid composite has a higher tensile strength than an untreated or a 10% treated fiber hybrid composite. This is due to the lack of removal of impurities from the fibers, which causes weak fiber matrix interfaces and a greater optimal condition for delignification, which causes weakening and damage to the fibers [13]. The 5% NaOH result demonstrates better tensile strength in both fiber-oriented composites. As the untreated (control) fibers have impurities that can affect the fiber-matrix interface and higher alkali concentrations (10% NaOH) can damage the fibers, the optimal setting for eliminating impurities from fibers is a 5% NaOH treatment. The woven types of fiber-orientation composites have higher tensile strengths than unidirectional fiber-orientation composites in the cases of untreated, 5%, and 10% NaOH-treated. The tensile strength of 5% NaOH-treated woven was improved by 5.2% compared with untreated enset/sisal composites. Low tensile strength due to poor adhesion between the matrix and the reinforcement.

3.2. Flexural Strength Test

The flexural test was performed by the three-point bending method according to ASTM D 790. Three specimens were tested, and the averages were calculated. The maximum load was applied in the middle of the specimen when it was freely supported by a beam. As shown in Figure 5, the woven type composites have higher flexural strengths than unidirectional composites. When 5% NaOH-treated woven composites were compared to untreated composites, their flexural strength increased by 9.2%. The adhesion between woven types of enset/sisal hybrid composites is better than that of unidirectional types of hybrid composites.

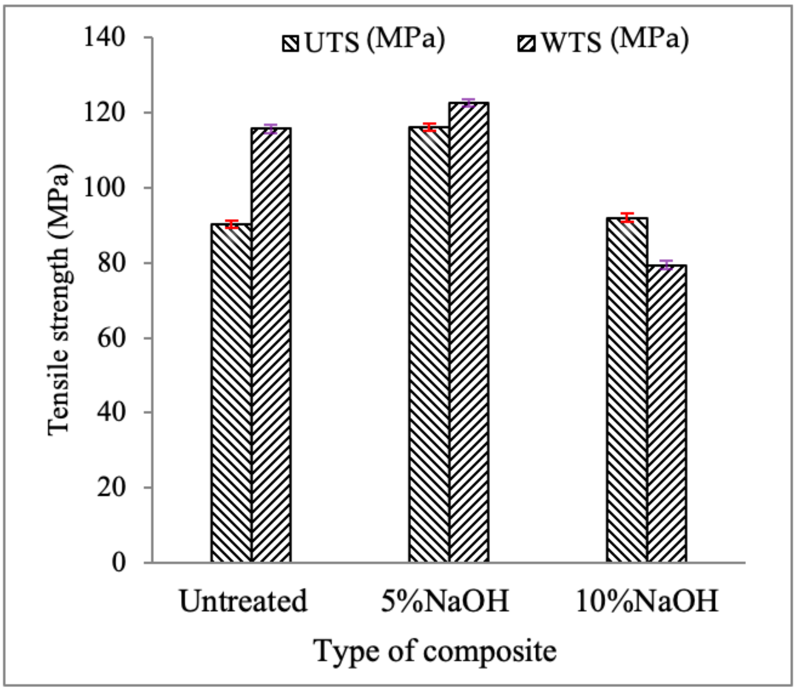

Figure 4. Tensile strength plot of unidirectional (UTS) and woven (WTS) composites.

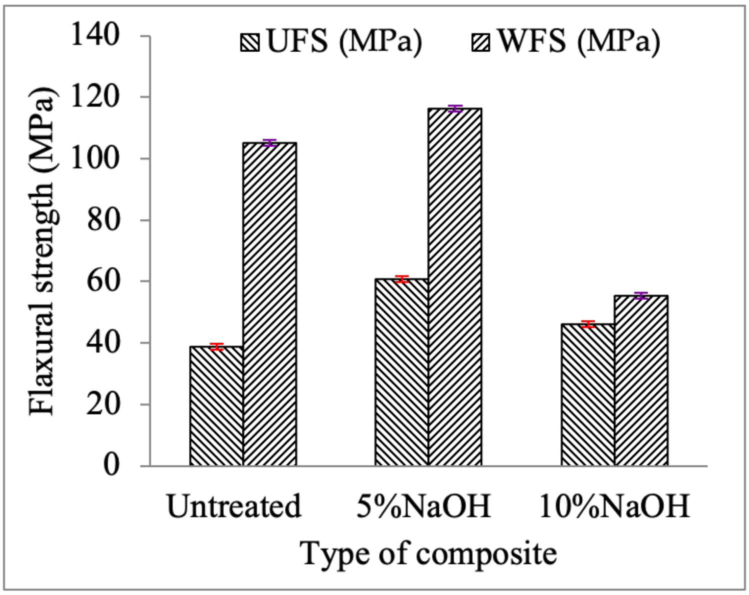

Figure 5. The flexural strength of unidirectional (UFS) and woven (WFS) composites.

3.3. Impact Strength Test

The cause of impact failure occurs when matrix fracture, fiber/matrix deboning, and fiber fracture are present in composite materials. Among these fibers, pull-out is found to be an important energy dissipation mechanism in fiber-reinforced composites [27]. Figure 6 shows that the 5% NaOH-treated enset/sisal hybrid composite has demonstrated a significant increase in the values of impact strength as compared to the untreated enset/sisal hybrid composite, which has shown an impact strength of 24.17 kJ/m^2. When compared to untreated composites, the impact strength of the unidirectional 5% NaOH-treated composites improved by 4.80%. When the woven 5% NaOH-treated composites were compared to the untreated composites, their impact strength increased by 5.98%.

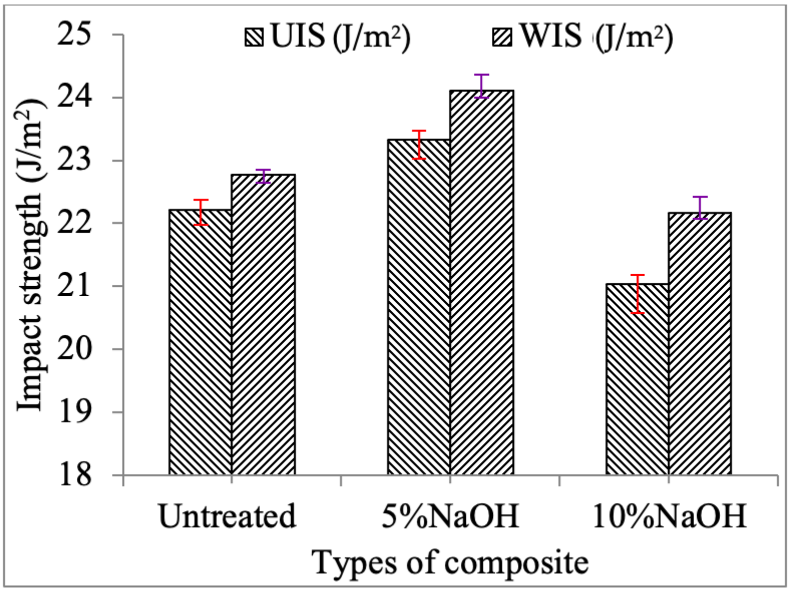

Figure 6. The impact strength of unidirectional (UIS) and woven (WIS) composites.

Although a strong binding must be guaranteed to produce materials for interior and structural applications, a weak interfacial link increases toughness by encouraging pullout effects required for energy absorption or impact strength purposes. For this reason, the 5% NaOH-treated composite has greater impact strength than the 10% NaOH-treated and untreated hybrid composites.

3.4. Water Absorption Test

As illustrated by the plots in Figure 7, all composites have the same water absorption characteristics, which implies that during the first 12 h, the range of water absorption varied from 5% to 6%. Then it climbed from 6.5% to 9% after 48 and 72 h and remained mostly unchanged after 84 h. When used as reinforcement in composite materials, the fiber's expansion upon water absorption would cause microcracks in the material, which would damage its mechanical qualities [28]. One can only draw the conclusion that fiber orientation, fiber treatment, and matrix compatibility can help natural fiber composites absorb less water. Compared to untreated fiber composites, treated fiber composites absorb less water, while treated woven composites absorb less water than treated unidirectional composites. Compared to untreated fiber composites, treated fiber composites absorb less water. On the other hand, treated unidirectional composite absorbs less water than treated weaved composite. This shows that better fiber-matrix compatibility leads to improved mechanical and water absorption properties.

3.5. Morphological Study of Composites

The ruptured surfaces of the fabricated composites after tensile testing were evaluated using SEM. We observe that the SEM images of untreated, 5% NaOH, and 10% treated enset/sisal hybrid composites are given in Figure 8a–c for woven and in Figure 9a–c for unidirectional, respectively. The images show that better bonding was observed at 5% NaOH-treated enset/sisal hybrid composites for both types of fiber orientations. But for untreated and 10% NaOH-treated samples, the SEM images show that there was large breakage of fibers and the presence of voids due to fiber pullout. This shows that adhesion between the fiber and the polyester as a matrix exists, which affects the mechanical properties of the composite.

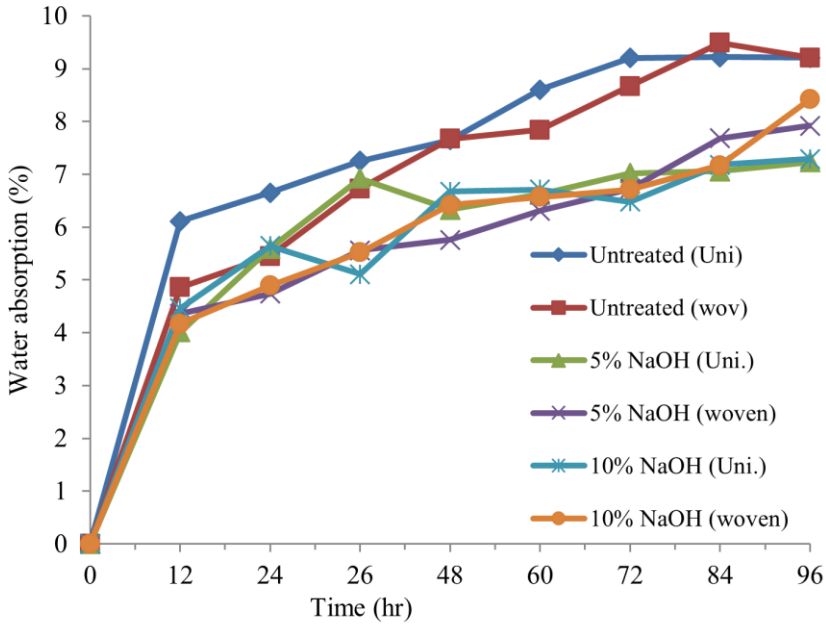

Figure 7. Water absorption test result.

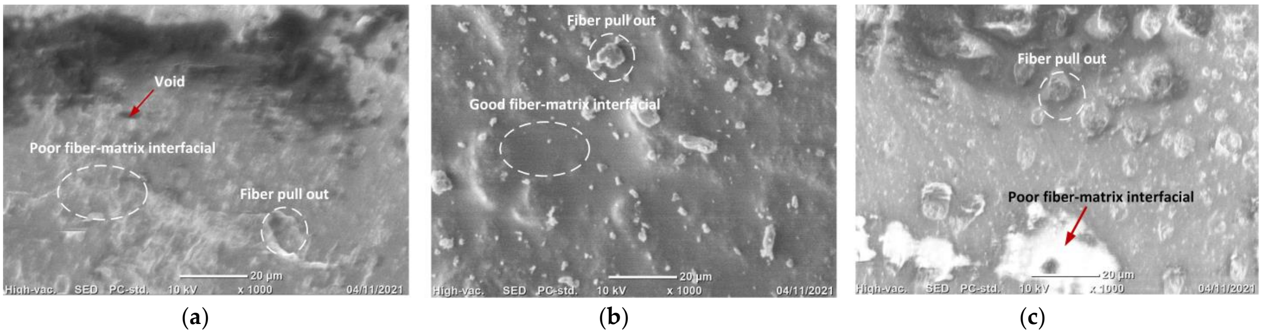

Figure 8. SEM image of woven enset/sisal hybrid composite (**a**) untreated; (**b**) 5% NaOH-treated; and (**c**) 10% NaOH-treated.

Table 1 shows a comparison of the mechanical properties (tensile, flexural, and impact) of untreated and treated (5% NaOH) samples, as well as a comparison with results reported in the existing literature of hybrid polyester composites. As we understood from the literature, treated hybrid composites have better mechanical properties than untreated hybrid composites, where treatment has improved the mechanical properties to a large extent. Similar results were also obtained in our study.

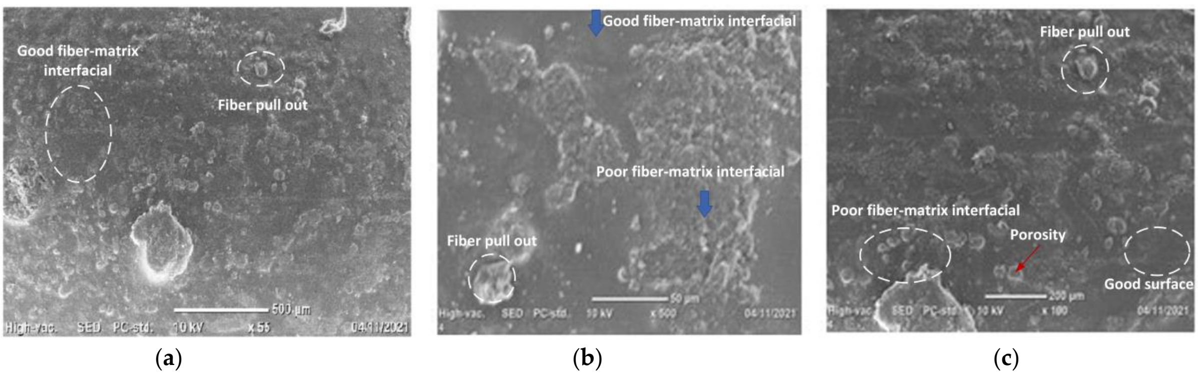

Figure 9. SEM image of unidirectional enset/sisal hybrid composite (**a**) untreated; (**b**) 5% NaOH-treated; and (**c**) 10% NaOH-treated.

Table 1. Effects of alkali treatment on mechanical behaviors of natural fiber-reinforced hybrid composites.

Hybrid Composites	Tensile Strength (MPa)		Flexural Strength (MPa)		Impact Strength		Reference
	Unt. *	Trt.	Unt.	Trt.	Unt.	Trt.	
Enset/Sisal unidirectional	90.23	116.12	38.77	60.92	22.21 J/m^2	23.33 J/m^2	Current Study
Enset/Sisal Woven	115.67	122.56	105.23	116.30	22.77 J/m^2	24.11 J/m^2	Current Study
Jute/Sisal	70.39	74.78	67.56	54.67	332 J/m	588 J/m	[11]
Jute/Curaua	66.77	63.9	97.67	80.86	388 J/m	288 J/m	[11]
Sisal/Jut	42.45	53.7 4	39.8	44.58	19.5 J	22.25 J	[29]
Bagasse aliphatic	23.47	26.77	43.87	50.86	8.82	11.27	[30]
Abaca epoxy	717	773	-		-		[31]
Sugarcane leaves/almond shell	12.86	17.16	39.91	40.68	0.78 J	1.05 J	[16]
sisal fiber epoxy composite unidirectional	132.73		288.6				[25]

* Note: Unt. = Untreated, Trt. = Treated.

3.6. Promising Application Area of E/S Composite

In comparison to other hybrid fiber-reinforced polymer composites, the 5% alkali-treated enset/sisal hybrid composites have comparable, acceptable, and satisfactory mechanical and physical properties that govern some promising and potential applications with the advantages of the environment, renewable resources, availability, and sustainability. Automotive, construction, aerospace, packaging, furniture, shipping pallets, electronic, medical, sporting goods, etc. are some of the most likely industrial applications. Concerns about cost, weight, and the environment prompted the use of composite materials made from natural fibers.

4. Conclusions

This study presented the effects of fiber alkali treatment and fiber orientation on the mechanical and water absorption properties of E/S hybrid composites. We draw those conclusions from the result.

- The mechanical properties of the hybrid composite are dependent on the interfacial bond between the fiber and matrix and on each lamina. So, fiber is one mechanism to improve the interfacial bond between fiber and matrix. The result of the study showed that treated fiber composites have better mechanical properties than untreated fiber hybrid composites.
- The woven type of fiber orientation has better mechanical properties than unidirectional fiber orientation in both untreated and treated hybrid composites.
- Tensile and flexural strengths of a 10% NaOH composite are similar to those of untreated fibers in unidirectional fiber orientation composites but less so in woven fiber orientation composites.
- The higher tensile and flexural strengths were obtained for 5% NaOH in both unidirectional and woven types of fiber orientation composites when compared with untreated and 10% NaOH-treated composites.
- The water absorption properties of treated E/S hybrid composites are less than those of untreated hybrid composites.
- The analysis of SEM images for unidirectional and woven types of composites revealed that the mode of failure of natural fibers and matrix during the tensile test was analyzed, and better surface interference was observed in woven fiber orientation composites with 5% NaOH treatment.

The E/S hybrid polyester composite was successfully fabricated, and the 5% NaOH treatment is used to improve the composite's properties for a wider range of applications.

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