



Article Investigation of Water Absorption Properties of 2D Interwoven Kevlar–Jute Reinforced Hybrid Laminates

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Abstract: The hygroscopic properties of natural fibers tend to degrade the mechanical properties of composite materials. It is essential to investigate the influence of water absorption behavior on the mechanical properties of hybrid composite materials. In this study, hybrid laminate materials consisting of two different reinforcement materials, i.e., Kevlar fibers and jute fibers in the same layer, are considered. Hybrid laminates that have four different weaving patterns: plain weave, basket weave, twill weave, and the satin weave, are tested for their water absorption characteristics. The jute fiber is a naturally extracted fiber that is subjected to chemical treatment. A comparison of mechanical properties before and after treatment of the jute fibers is carried out. Mechanical properties such as tensile strength, compressive strength, flexural strength, impact strength, and hardness are tested. It is found that the mechanical properties improved after the treatment of the jute fibers. The twill weave pattern exhibited negligible defects compared to its counterparts. Chemical treatment of the jute fibers is mproved after using chemically treated jute fibers as the reinforcement material in the hybrid laminate materials. It is concluded that the twill weave pattern and chemical treatment of the natural fibers improved the mechanical properties of the hybrid laminate materials.

Keywords: mechanical properties; water absorption behavior; jute fibers; Kevlar fibers; hybrid laminates

1. Introduction

Hybrid laminate materials are a class of composite materials that comprise two or more different fibrous materials as their reinforcement and a polymer as the matrix element [1]. Hybrid laminate materials are preferred in applications such as automobile bodies, locomotive body panels, small boats, building components, and aircraft panels [2,3]. They offer superior strength and low weight compared to conventional materials and simple composite materials [4–6]. The properties exhibited by every composite material depend on factors such as the property of the individual components, the composition of the materials, the manufacturing methods adopted, the orientation of the reinforcement fibers, pre-processing and post-processing techniques, and the presence of defects [7–10].

The literature reveals that synthetic fiber materials such as Kevlar, glass fibers, and carbon fibers exhibit superior tensile strength, low thermal conductivity, low electrical conductivity, and low density [11–14]. These properties make them preferable for high-end applications such as aircraft structural materials, sports cars, marine components, building materials, and rockets. However, a few drawbacks, such as high cost, high brittleness, difficulty to procure, and hazard to the environment, make these synthetic materials a poor contender for domestic and commercial applications [15,16].

Natural fiber materials such as kenaf, areca, jute, coir, animal hair, and basalt offer benefits that are not exhibited by their synthetic counterparts. Despite the many advantages



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). of natural fibers, they struggle to compete with synthetic fibers because of a few major drawbacks. Natural fibers have poor tensile strength and are prone to degradation because of their hygroscopic properties [17–21]. Many literature works have attempted to combine the properties of natural and synthetic fibers. When combining natural and synthetic fibers, the beneficial properties, as well as the ill effects of the respective materials, are added to the produced hybrid composite materials [22–24].

Many works of literature have highlighted the benefits of Kevlar fibers on the produced composites and hybrid composite materials [25–27]. Likewise, the influence of natural fibers such as jute on the hybrid laminates has been well documented [28–32]. Patil et al. (2017) [33] carried out a study in which they discussed the characterization of Kevlar–jute hybrid laminate materials. They found that increasing the thickness of the hybrid laminate from 2 mm to 4 mm resulted in reducing the tensile strength and flexural strength by 1.5% and 63.5%, respectively. However, the compressive strength increased by 55%. The number of layers in the hybrid laminates played a dominant role in the mechanical properties exhibited by the hybrid laminate.

Several works of literature have discussed the influence of the stacking sequence on the mechanical properties of hybrid laminate materials. A test was conducted on kenaf-areca hybrid composite materials with 10 wt.% of wood dust filler, and the material absorbed impact loads ranging from 14 J to 18 J before fracture [34]. However, without the filler materials, the impact energy of the hybrid composite before failure increased to 46 J and a maximum tensile strength of 17.26 MPa [35]. Combining a synthetic fiber, i.e., Kevlar, with a natural fiber, i.e., basalt, resulted in a tensile strength of 174 MPa, a flexural strength of 188 MPa, and an impact energy of 24 J [36,37]. Similarly, a hybrid laminate material containing carbon and jute as the reinforcement materials exhibited a tensile strength of 280 MPa [38]. Chemical treatment of natural fibers removes their hygroscopic properties [39–41]. This process benefits in enhancing the mechanical properties of the hybrid laminate materials. The impact energy absorbed by a hybrid laminate containing three different reinforcement fibers, i.e., carbon, abaca, and kenaf, increased from 14 J to 26 J after the chemical treatment of the natural fibers [42].

From the literature survey, it was found that the hygroscopic properties of the natural fibers diminish the mechanical properties of the laminate material. This is a drawback in composites that are produced using natural fibers as their reinforcement. Chemical treatments are suggested to remove the hygroscopic properties of the natural fibers. This improves the mechanical properties of the resulting laminate material. The literature works highlights the removal of hygroscopic properties by comparing the mechanical properties. However, a discussion on the microstructural behavior of the laminate material can give an insight into their water absorption characteristics. This study compares the mechanical properties is laid one over the other. This method is called stacked reinforced laminates. However, this study focusses on novel hybrid laminates in which the two reinforcement materials are interwoven in the same layer.

A new class of reinforcement materials containing two different materials interwoven along the waft and weft direction has emerged recently [43,44]. However, very few articles have covered the influences of interwoven fibers on the mechanical and microstructural properties of hybrid laminate materials. In this study, a 2D interwoven hybrid composite consisting of two different reinforcement fibers, i.e., Kevlar and jute, is considered. The hybrid laminate is fabricated using a Vacuum Assisted Resin Transfer Molding (VARTM) method. The natural fiber, i.e., jute, is subjected to chemical treatment to remove its hygroscopic properties. The influence of weaving pattern on their mechanical and microstructural properties are studied. The results thus obtained are compared with their counterparts with non-treated fibers.

2. Materials and Methods

2.1. Selection of Materials and Weaving Patterns of Reinforcement Fibers

Reinforcement fibers comprising Kevlar and Jute were twinned to a diameter of 1 mm. They were interwoven to a mat in which the Kevlar fibers were laid along the warp direction and the jute fibers were laid along the weft direction. Four different weaving configurations were used for this study: plain weave, basket weave, twill weave, and satin weave, as shown in Figure 1. The thickness of each mat was 1.5 mm.



Figure 1. Weaving configuration of the reinforcement fibers. (a) Plain weave; (b) Basket weave; (c) Twill weave; (d) Satin weave.

2.2. Treatment of Fibers

Two different sets of reinforcement fibers were used for the study. One contained untreated fibers, while the other set contained treated jute fibers. A solution containing 5 vol.% of NaOH dissolved in water was prepared [16]. The woven fiber mat was then immersed in the solution. After 3 h, the fibers were removed from the solution and allowed to dry under sunlight for two days. The chemical reaction that takes place during the treatment of the fiber is shown in Equation (1). The resulting natural fiber threads with their hydrophilic nature removed were washed in distilled water to remove the traces of sodium hydroxide (NaOH). The treatment with NaOH removes the lignin and pectin present in natural fibers. Further, it prevents the growth of pathogens in the fibers.

Natural Fiber + NaOH
$$\leftrightarrow$$
 O - Na - Fiber Threads (1)

where NaOH is Sodium Hydroxide, O is oxygen, and Na is Sodium.

2.3. Production of Hybrid Laminates

The hybrid laminates for this study were produced using the VARTM method. The mold frame had a square area of 0.25 m^2 , and the woven mats were each cut to $500 \text{ mm} \times 500 \text{ mm}$. The first hybrid laminate was produced by stacking three plain weave mats with a 1 mm gap between the adjacent layers. The binding material was prepared by mixing the resin (LY556) and hardener (HY951), mixed in a ratio of 10:1 in a separate container connected to the inlet of the mold through a pump capable of delivering 2 bar pressure. The air inside the mold was removed using 0.966 bar pressure from a vacuum pump before applying the binding material [45].

The filling of the binding material was ensured when the excess was spilled out through the discharge valve from the mold. The composition of the reinforcement fibers and the binding material was maintained in a volume composition of 40:60 [37]. The mold was transferred to an oven to cure the contents at 80 °C for 45 min. It was then cooled under ambient conditions before removing the resulting hybrid laminate, as shown in Figure 2. Eight different hybrid laminates were produced for the study. The thickness of each hybrid laminate was approximately 5 mm. Four of the hybrid laminates contained untreated fibers in varying weaving configurations. The other four contained treated fibers as their composition.



Figure 2. Hybrid laminated.

The density of the produced hybrid laminate material was found using Equation (2). The mass of the laminate is determined using Equation (3).

$$\rho_{\rm l} = \sum_{1}^{\rm n} \frac{W_{\rm n}}{\rho_{\rm n}} \tag{2}$$

where ρ_1 = density of the hybrid laminate, n = composition, W = weight fraction of the respective composition, and ρ_n =density of the respective composition.

$$M_1 = \rho_1 \times V_1 \tag{3}$$

where M_1 = mass of the hybrid laminate and V_1 = volume of the hybrid laminate.

The mass of the binding material is found by multiplying 0.6 by M_1 . This is because the binding material, i.e., the matrix element, consists of 60% of the total mass of the hybrid laminate, whereas the reinforcement comprised 60% of the total mass of the hybrid laminate. Thus, the reinforcement to the matrix element was maintained as 40:60. The mass of the jute and Kevlar fibers is found by multiplying the mass of the individual fiber sample by three. This is because each hybrid laminate consists of three layers of interwoven jute and Kevlar fiber mat. The void fraction is the same as the density of the produced hybrid laminate material [46].

Table 1 shows the void fraction of the hybrid laminate materials produced for this study. It can be noted that the theoretical and actual density of the hybrid laminate materials varies concerning the weaving pattern. This phenomenon occurs because of the increase in the quantity of epoxy resin in the respective hybrid laminate material. The maximum void fraction was noted in the case of the satin weave pattern, whereas the lowest void fraction was noted in the twill weave pattern.

S. No.	Laminate Material	Theoretical Density (g/mm ³)	Actual Density (g/mm ³)	Void Fraction (%)
1	Plain weave	105.8	44	0.584
2	Basket weave	114.5	51.5	0.55
3	Twill weave	115.7	58.7	0.49
4	Satin weave	111.3	43.95	0.605

Table 1. The void fraction of the hybrid laminate materials.

2.4. Testing the Mechanical Properties and Microstructural Examination

The produced hybrid laminate materials containing different weaving patterns were tested for their mechanical properties. The tensile strength, flexural strength, compressive strength, impact strength, and hardness were tested. For each test, five specimens were cut as per ASTM standards. ASTM D3039 for the tensile test, ASTM D 6641 for the compressive test, ASTM D790 for the flexural test, and ASTM D256 for the impact test were used. The five sets of readings for each test were used to find the error percentage for each mechanical property. The test specimen that was cut as per the ASTM standards were warmed inside a muffle furnace at 45 °C for 24 h to remove the moisture present in them.

The tensile test was carried out using a universal testing machine (Model: Instron-UNITEK-94100). The test specimen was held between the jaws of the universal testing machine and subjected to tensile load at 5 mm/min. The resulting tensile strength was recorded. In the same manner, the compressive strength of the hybrid laminate materials was recorded. The flexural test was carried out using the three-point bending load approach. The test specimen was given a shear load at the midpoint by moving the jaw at 5 mm/min. The Izod impact strength was recorded for the hybrid laminate materials. The shore D hardness value was measured for each test specimen. Microscopic examinations were carried out on the hybrid laminates to study the characteristics of the reinforcement and epoxy resin. For this, Scanning Electronic Microscope (SEM) images were taken at the resolution of $200 \times to 1000 \times$.

2.5. Water Absorption Test

Samples were prepared from the eight hybrid laminates based on ASTM D570-98 standards. Each specimen measured 76 mm \times 25 mm. Distilled water was used during the test. Each specimen was first wiped using a clean cloth to remove dirt and dust. It was then heated to 45 °C for 30 min to remove any moisture present in it. The water absorption test was carried out in batches. One batch consisted of untreated hybrid laminates, as shown in Figure 3. The other batch consisted of their respective counterparts with treated fibers.







The hybrid laminates in each batch were allowed soak for 24 h. They were then briefly taken out and wiped using a clean cloth to remove surface water. The weight of the specimen was measured at room temperature using a digital weighing machine before immersing it back in distilled water [39]. Equation (4) shows the formula to measure the percentage change in water absorbed by the respective specimen. The moisture absorption was found for each hybrid laminate material for seven weeks, i.e., 49 days.

$$W_a = \frac{\text{Difference in the weight of the Sample}}{\text{Actual weight of the sample}} \times 100$$
(4)

where W_a is the percentage of water absorbed by the test specimen.

3. Result and Discussion

3.1. Microscopic Analysis

Figure 4 shows the Scanning Electron Microscope (SEM) images taken for the four different hybrid laminates. In the case of the plain weave pattern, the Kevlar and jute fibers laid along the waft and weft direction were misoriented, as shown in Figure 4a. The improper orientation of the fibers in the SEM image reveals that the epoxy resin flow was violent when they were introduced through the VARTM. This caused the epoxy resin to flow unevenly over and into the fibers in the warp and weft directions. Because of this, microvoids were noted between the fibers.

The size of the voids was even and distributed along the area of the fiber mat. The uneven flow of the epoxy resin resulted in the resin forming lumps on the surface of the produced hybrid laminate. The basket weave pattern enabled a fairly uniform distribution of the epoxy resin, as shown in Figure 4b. Compared to the plain weave, the epoxy resin was noted to disperse along the surface of the produced hybrid laminate. However, the microvoids were still present in the hybrid laminate. The number of microvoids in the basket weave configuration was less compared to that present in the plain weave configuration. This implies that the epoxy resin had comparatively better bonding with the reinforcement fibers. The twill weave pattern allowed the epoxy resin to flow evenly and disperse properly along the Kevlar fibers and jute fibers. As a result, there was superior bonding between the epoxy resin and the reinforcement fibers in the warp and weft directions.

50 µm

SRM IST-Apreo S



Figure 4. Cont.

use case

use case 5/28/2021 det HV WD pressure mag □ dwell spot srot Standard 3:09:27 PM LVD 10.00 kV 11.1 mm 20 Pa 1000 x 3.00 µs 4.0 0°

(b)



Figure 4. SEM images of the hybrid laminates. (**a**) Plain weave pattern; (**b**) Basket weave pattern; (**c**) Twill weave pattern; (**d**) Satin weave pattern.

A relatively thick layer of epoxy resin formed on the surface of the produced hybrid laminates. Because of this, the fibers were not visible in the SEM image shown in Figure 4c. The satin weave configuration interfered with the flow of epoxy resin during the VARTM process. As a result, the epoxy resin dispersed unevenly in the warp and weft directions of the Kevlar and jute fibers. Segments of epoxy resin patches can be observed in the SEM image shown in Figure 4d. Relatively large microvoids occurred due to the improper flow and settlement of the epoxy resin over and into the reinforcement fiber of the satin weave configuration.

3.2. Water Absorption Test

Comparison is made between the four hybrid composites with untreated jute reinforcement fibers, as shown in Figure 5. It is observed that out of the four different hybrid composites containing different weaving patterns, i.e., plain weave, basket weave, twill weave, and satin weave, the plain weave pattern absorbed the maximum water. Within seven days of the water absorption test, the plain weave hybrid laminates absorbed up to 23.5% water. However, the twill weave pattern absorbed only 15.8% water during the same interval. The other two weaving patterns, i.e., basket weave and satin weave, absorbed 21.7% and 17.4% water, respectively. The weaving pattern influenced the water absorption capability of the hybrid laminates. The twill weave pattern has tightly packed fibers compared to its counterparts, whereas the plain weave pattern has the most space between the fibers among its counterparts.



Figure 5. Untreated hybrid laminates in distilled water.

During the water absorption test, it was noted that during the fifth week, the two weaving patterns, i.e., basket weave and satin weave, exhibited a greater water absorption rate, i.e., an increase of 1.1% and 2.4%, respectively. This is because the epoxy resin developed voids in the middle layers of the reinforcement fibers. It took five weeks for the moisture to seep into the voids that were hidden in the middle layers of the reinforcement. In the case of the plain weave and twill weave pattern, the water absorption was increasing steadily throughout the test. However, until the end of the test, i.e., the 49th day, the twill weave pattern maintained a very low water absorption of 23.3%. However, the plain weave pattern exhibited the highest water absorption of 30%.

Figure 6 shows the comparison made between the four hybrid composites with treated jute reinforcement fibers. Also during this comparison, the plain weave pattern exhibited very high-water absorption compared to the other three weaving patterns. This justifies the claim that the plain weave pattern has sufficient space between the fibers, which allowed

water to seep in. However, unlike the untreated jute fibers, the treated jute fibers discharged excess moisture after the fifth week. This phenomenon occurred because the treated jute fibers did not have hygroscopic properties. As a result, the water absorption occurred only because of the voids between the epoxy resin and the reinforcement fibers.



Figure 6. Treated hybrid laminates in distilled water.

In the other three weaving patterns, the water absorption reduced after the fourth week. It was noted that after the fourth week, the water absorption in the basket weave and satin weave was less than in the twill weave pattern. This phenomenon occurred because the twill weave pattern did not have any voids within the epoxy resin and the reinforcement fibers. The water that was absorbed by the basket weave pattern and the satin weave leaked through the voids as they were cleaned during the test. The same phenomenon is also observed in the plain weave pattern.

3.3. Mechanical Properties of the Hybrid Laminates

The mechanical properties such as tensile strength, compressive strength, flexural strength, impact strength, and hardness, were determined. During the testing of the hybrid laminate containing untreated fibers, it was found that the weaving patterns influenced the mechanical properties [47]. Table 2 shows the mechanical properties obtained from a hybrid laminate with untreated jute fibers. The plain weave pattern exhibited very low mechanical properties compared to the other three weaving patterns. This is because the hybrid laminate material containing the plain weave pattern has considerable microvoids in it. The microvoids develop extensive internal stress that contributes to the limited load-bearing capacity of the hybrid laminate material. The twill weave pattern exhibited superior mechanical properties. This phenomenon is associated with the proper bonding between the matrix element and the three layers of reinforcement fibers. This facilitated an increase in the load-bearing capability of the hybrid laminate material. The impact strength of the hybrid laminate with the satin weave pattern was the lowest. This is because the microvoids in these hybrid laminates were large. During the impact test, the internal stress developed closer to the microvoids, causing shear. This shear gave rise to a brittle fracture of the matrix element. Because of this phenomenon, the hybrid laminate ruptured easily compared to its counterparts with the other three weaving patterns.

Hybrid Laminate	Tensile Strength (GPa) \pm SD	Compressive Strength (MPa) \pm SD	Flexural Strength (GPa) \pm SD	Impact Strength (J) \pm SD	Shore D Hardness \pm SD
Plain weave	1.95 ± 2.045	52.8 ± 3.1	2.4 ± 1.07	20 ± 2	68.7 ± 1.1
Basket weave	3.65 ± 2.08	62 ± 2.4	3.12 ± 2.04	61 ± 2	65.7 ± 1.3
Twill weave	6.77 ± 2.09	105.6 ± 3.6	3.4 ± 1.06	89 ± 3	72.2 ± 1.4
Satin weave	2.91 ± 1.09	88.8 ± 2.5	3.6 ± 2.07	15 ± 1	71.5 ± 1.3

Table 2. Mechanical properties of hybrid laminates with untreated fibers.

Table 3 shows the mechanical properties measured from the hybrid laminate materials with chemically treated jute fibers. It was found that the chemical treatment and the weaving pattern played a vital role in the characteristics of the hybrid laminate materials. The tensile strength of the plain weave hybrid laminate increased from 6.85% in the basket weave pattern to 12.25% in the twill weave pattern. The increase in the tensile strength shows that the chemical treatment of the jute fiber effectively removed its hygroscopic properties. The highest tensile strength is exhibited by the hybrid laminate with a twill weave pattern. It is implied that the superior bonding between the matrix element and the reinforcement fibers in the hybrid laminate with a twill weave pattern facilitated the superior distribution of the applied load. This enhanced the tensile strength of the respective hybrid laminate material. However, the presence of microvoids in the hybrid laminate with plain weave and satin weave patterns hindered the load distribution during the tensile test. Because of this, they exhibited less tensile strength.

Fable 3. Mechanical properties of hybrid laminates with treated fibers.
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Hybrid Laminate	Tensile Strength (GPa)	Compressive Strength (MPa)	Flexural Strength (GPa)	Impact Strength (J)	Shore D Hardness
Plain weave	2.1 ± 1.05	55.2 ± 3.5	2.9 ± 1.04	21 ± 1	68.8 ± 1.3
Basket weave	3.9 ± 2.06	65 ± 3.45	4.2 ± 2.05	63 ± 2	65.5 ± 1.2
Twill weave	7.6 ± 2.5	111.3 ± 3.61	4.8 ± 2.15	91 ± 2	72 ± 1.4
Satin weave	3.14 ± 2.05	91.6 ± 2.38	4.5 ± 1.05	17 ± 1	71.3 ± 1.3

The compressive strength and flexural strength measured from the hybrid laminates with treated jute fibers exhibited a similar trend to that obtained from the tensile test. The compressive strength after the treatment of the jute fibers increased from 3.15% in the satin weave pattern to 5.4% in the twill weave pattern. Likewise, the flexural strength after the treatment of the jute fibers increased from 21% in the plain weave pattern to 41% in the twill weave pattern. It can be observed that the treatment of the jute fiber altered the mechanical properties exhibited by the hybrid laminate materials.

Comparing the hybrid laminates with treated jute fibers and their counterparts with untreated fibers shows that they exhibited a similar impact strength and hardness. During the impact test, the presence of microvoids controlled the distribution of shear load. However, the weaving pattern at the surface of the hybrid laminate materials affected the hardness value. It was found that the hybrid laminate with the twill weave pattern exhibited superior mechanical properties compared to the other weaving patterns.

4. Conclusions

Hybrid laminates with four different weaving patterns, i.e., plain weave, basket weave, twill weave, and satin weave, were produced using the vacuum-assisted resin transfer molding method. The hybrid laminates consisted of reinforcement materials with Kevlar fibers in the warp direction interwoven by jute fibers in the weft direction. Each hybrid laminate material considered for this study had three layers of interwoven reinforcement fibers bonded with epoxy resin. Two sets of hybrid laminates were considered for the study, in which one set had untreated jute fibers, while the other set had the jute fibers treated with 5 vol.% of sodium hydroxides in distilled water. Microscopic examination revealed microvoids in the hybrid laminate materials with the plain weave and the satin weave patterns. Treated jute fibers absorbed less water because the chemical treatment removed their hygroscopic properties. Mechanical examination revealed that the tensile strength, compressive strength, and flexural strength increased after using treated jute fibers as the reinforcement composition. The impact strength and hardness did not vary, irrespective of the treatment of the jute fibers. When compared to the four different weaving patterns in the reinforcement, the twill weave pattern exhibited a superior tensile strength of 7.6 GPa, a high compressive strength of 111.3 MPa, and a high flexural strength of 4.8 GPa. The impact energy absorbed before failure in the same hybrid laminate materials was 91 J, and the shore D hardness value was 72. It is concluded that the chemical treatment of jute fibers enhances the mechanical properties of the hybrid laminate materials, and the twill weave pattern develops fewer defects, which facilitates enhanced mechanical properties.

This hybrid laminate is suitable for producing components and parts that are required to withstand forces with high mechanical properties. Parts such as automobile bonnets, automotive dash boards, locomotive partition boards, marine structures, aircraft panels, building materials, and defense materials can be produced using the suggested hybrid laminate.

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