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AN EXPERIMENTAL INVESTIGATION OF THE EFFECT OF NATURAL FIBER TREATMENT AND MARINE ENVIRONMENT ON CANNABIS SATIVA/EPOXY LAMINATES

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Materials have helped in evolving technology to a great extent. Composites have replaced conventional metals/ non-metals because of their lightweight. Natural Fibers have been need of the hour owing to environmental concerns and ease of availability. In this work, Cannabis Sativa fibers were treated with 5% Potassium Hydroxide solution. The laminates were prepared by the Compression Moulding technique by reinforcing treated and untreated fibers with an epoxy matrix material. To access the durability of natural fiber composites in the marine environment, prepared laminates were aged in seawater for 150 days. Tensile, flexural and moisture absorption behavior tests have been performed to estimate the durability in seawater. The data obtained have been compared with pristine treated and untreated fiber reinforced samples. From the results, it has been observed that tensile and flexural behavior of untreated fiber reinforced composites were superior to a treated counterpart in both pristine and aged conditions.

Key words: treated, untreated, ageing, sea-water, modulus

INTRODUCTION

Composite contains two important components namely the fiber and matrix phase. The role of fiber is to make them stronger, firm and bears the load. The property of the matrix is that it is consistent, holds the fiber intact and provides shape to the structures [1]. Fiber and matrix can distinguished one from the other in composite material but together they exhibit properties that cannot be attained by individual material [2]. Natural fibers are usually obtained from either plants or animals[3],[4]. They can be easily recycled and hence environmentally friendly [5], [6]. Some other benefits of using natural fiber over synthetic fibers include reasonably priced, low weight, available in large quantities, negligible damage environment [7]. Some of the most common techniques to extract fibers from plants is by decortication and retting. In retting, the woody part of stem undergoes biological treatment which results in the fibers falling apart whereas in decortication a machine called decorticator pulls out the fibers from the leaf or stem [8–10].

Cellulosic fibers of plants can be categorized into bast, seed, leaf, reed, grass and core fibers. Some examples of bast fibers include jute, flax, hemp, ramie, and kenaf [11]. However, these natural fibers have some minor setbacks these include moisture uptake and also improper adhesion with the matrix material. But this can be addressed by subjecting to specific surface treatments of which reduces the water intake together with improvement incompatibility between fiber and matrix [12], [13]. The mechanical conduct of Natural Fiber Reinforced Composites (NFRCs) depends on various parameters these include fiber orientation, moisture absorption, impurities, physical properties, and volume fraction [14], [15], [16], [17], [18], [19].

Cannabis Sativa or hemp has been grown and harvested once a year. The seeds and fibers of hemp are in most demand. Cultivation of this crop was banned in most developed countries due to its usage as an illicit drug[20]. These fibers have a wide variety of applications such as in agriculture, textile, bio-composite, paper-making, automotive, construction, biofuel, functional food, oil, cosmetics, personal care, and pharmaceutical. The woody portion of the hemp plant and tissues have more lignin can be utilized for bedding of horses, pulping and concreting [21]. Presence of more cellulose and also since it is cultivated on a large scale hence it can also serve as biomass [22]. The rise of demand for such crops is due to more yield combined with viability [23].

The present research work is focused to understand the effect of fiber treatment on tensile and flexural behavior of Hemp/Epoxy laminates. The study also focuses to evaluate the durability of natural fiber-reinforced composites in the marine environment. Fibers were treated with 5% Potassium Hydroxide (KOH) solution and laminates were prepared by the compression molding process. The prepared laminates were aged in natural seawater for 150 days in ambient conditions. Fourier Transform Infrared Spectroscopy and Scanning electron microscope images were used to study the treatment effect on fiber and tensile failure surface morphology respectively.



Figure 1: Alkaline treatment of hemp fibers



Figure 2: Representation of the laminate preparation process

MATERIALS & METHODS

Materials

Bidirectional Hemp fiber (BF 31 A) with an aerial weight of 400 GSM were procured from Hemp Fabric Lab, Mumbai, India. The density of the procured hemp fabric was 1.48g/cm³. Laminates were prepared by reinforcing treated and untreated fibers with the thermoset matrix material. Atul grade Epoxy (Lapox L-12) with a gel time of 45 minutes and K-6 hardener were obtained from Yuje Enterprises, Bangalore, India.

Alkali treatment of fibers

In the present work, hemp fibers were treated with 5% KOH. Initially, the fibers were pre-washed with distilled water for 1 hour to clear the fibers from any dirt. Later the fibers were dipped in 5% KOH and kept in the solution for 2 hours as shown in Figure 1.

After the treatment fibers were washed with distilled water to remove any KOH bound to the fiber surface. The treated fibers were allowed to dry in ambient temperature for 48 hours.

Laminate preparation

The treated and hemp/epoxy and untreated hemp/epoxy examined in this study virtually identical by all means (Fiber weight fraction, Number of layers and fabrication method). The specimens were prepared by reinforcing 4 layers of bi-directional treated and untreated hemp fibers with the epoxy matrix material. Initially, two mild steel plates bearing dimensions of 300×300 mm is chosen and cleaned with acetone to remove any dirt on the surface. Later a coat of release agent is applied to both the plates. The laminate consisting of 4 layers of fibers and a mixture of resin and hardener in between each layer as well as a layer on either side of reinforcement was

prepared by hand lay-up technique. Finally, a sheet of peel ply bearing dimension of 300×300 mm was placed on this to prevent any foreign particles from entering it. To obtain consistent quality and equal thickness the prepared laminates were compressed using compression molding machine and schematic representation of the same as shown in Figure 2.

The compressed laminates allowed to cure in a pressed stage for 24 hours in ambient conditions. The average thickness of the prepared laminate was found to be 2.5 mm. The density of the specimens was measured using Archimedes principle. 30 specimens were used to obtain the average density and were found to be 1.368±0.16 g/cm³.

Accelerated ageing

In the field of civil engineering and marine industry, composite materials were exposed to different service conditions. To understand the interaction between seawater and natural fiber-reinforced composites the prepared laminates were immersed in seawater for 150 days in ambient conditions. Seawater required for the aging process was collected from the Arabian Sea at the western coastal region of Karnataka. Two rectangular (50×50×2.5 mm) specimens of untreated fiber-reinforced composites (UFRC) and treated fiber-reinforced composites (TFRC) were used to monitor the moisture absorption behavior of the laminates. Moisture concentration of the laminates during the ageing period in regular intervals monitored and expressed by using gravimetric Equation 1 [32, 33]. The initial weight and weight of the specimens in equal intervals monitored using 0.001 mg accuracy digital weighing scale.

$$M(t) = \left(\frac{W_t - W_i}{W_i}\right) \times 100$$
(1)

Where M (t) is the moisture content of the material at any time in %, W_i is the dry or initial weight and W_t is the wet weight of the samples.

Tensile test

The tensile properties of untreated fiber-reinforced composites (UFRC) and treated fiber-reinforced composites (TFRC) were investigated as per ASTM 3039 with a dimension of 250×25×2.5mm [32]. Tests were conducted using 50 kN BiSS make Universal Testing Machine with a crosshead speed of 2mm/min. To obtain the average failure stress and strain five replicates of specimens were used in each condition. The maximum failure stress and strain of each specimen were calculated from the data obtained.

Flexural test

The flexural test was carried out on UFRC and TFRC as per ASTM D 790-03 with the help of FIE make 50 kN Universal Testing Machine. Tests were conducted by maintaining a gauge length of 75 mm and a width of 13 mm. The maximum load at which it buckles is obtained



along with the displacement is noted. Flexural strength and modulus for every specimen is calculated from the data obtained.

RESULTS & DISCUSSION

Chemical Analysis

Fourier Transform Infrared Spectroscopy (FTIR) analysis was used to characterize and to compare the chemically modified hemp fiber with untreated hemp fiber. Figure 3 shows the infrared spectra in the range of 4000-500 cm-1 for untreated and treated hemp fiber. Untreated fiber showed the presence of hydroxyl group in the wavelength between 3500-3000 cm-1. But treated fiber showed elimination of hydroxyl group: which will reduce the affinity towards moisture. Also, 3000-2500 cm-1 indicates the C-H bond stretching for untreated hemp fibers. The treatment of fiber with KOH eliminated this group and indicates the removal of lignin from the fiber surface. At 1650 cm-1 shows the carbonyl group stretching which designates removal of hemicellulose [28], [34], [35], [36].

Moisture absorption behavior

Exposure of polymer matrix composites to humid or water environments will induce absorption of moisture into the material by a diffusion process. The Figure 4 shows the moisture absorption behavior of UFRC and TFRC laminates as a function of immersion time.

Surface modification of Hemp fabric with KOH reduced the water affinity of the fibers and resulted in a lower amount of moisture intake as compared to UFRC. During the aging process, both specimens exhibited two stages of moisture absorption phenomenon by following Fick's law of diffusion. In the initial immersion period, a higher rate of moisture uptake process was observed at 650 hours of immersion both specimens reached to saturation point or an equilibrium point. At saturation point, UFRC and TFRC absorbed 9 and 8% of moisture respectively as compared to their initial weight.



Figure 3: FTIR spectra of treated and untreated Hemp fiber





Figure 4: Moisture absorption behavior of UFRC and TFRC hemp/epoxy laminates

Tensile Test

The tensile properties of the composite material mainly depended upon the fiber/matrix bonding, fiber content, void content and moisture absorption rate. Table 1 and 2 shows the variation of tensile properties of untreated and treated Hemp/Epoxy laminates at different aging period. In each test 5 samples used to obtain the aver-

Table 1: Effect of ageing duration on tensile properties of UFRC hemp/epoxy laminate

Ageing duration (Days)	Average Tensile Stress (MPa)	Average Failure strain (mm/mm)	Average Modulus (GPa)
0	36.478±3.066	0.036±0.006	0.855±0.028
30	34.383±1.913	0.033±0.005	0.831±0.021
90	31.620±4.847	0.034±0.001	0.798±0.016
150	31.570±1.604	0.0355±0.002	0.766±0.029

Table 2: Effect of ageing duration on tensile properties of TFRC hemp/epoxy laminate

Ageing duration (Days)	Average Tensile Stress (MPa)	Average Failure strain (mm/mm)	Average Modulus (GPa)
0	32.246±0.831	0.043±0.004	0.754±0.032
30	28.556±0.985	0.036±0.008	0.721±0.017
90	25.916±2.642	0.034±0.008	0.685±0.037
150	23.248±2.149	0.035±0.004	0.681±0.037

age failure stress and strain of the material. The analysis of obtained results shows that UFRCs exhibited better stress-bearing capabilities compared to TFRCs.

Surface modification of fibers was generally used to improve the adhesion between the fiber/matrix interphase. Treatment of fibers with 5% KOH resulted in the removal of lignin and hemicellulose from the fiber surface. Also, the treated fibers become stiffer and it's mass also re-





Figure 5: Variation of failure stress of UFRC and TFRC with respect to ageing duration

duced in comparison to untreated ones [37], [38]. Figure 5 shows the variation of failure stress with respect to the aging period for both TFRC and UFRC specimens. In the case of a pristine specimen, TFRC specimens showed a 12% reduction in maximum failure stress as compared to UFRC. Treatment of fibers with a higher percentage of KOH resulted in poor interfacial strength and showed a reduction of tensile properties. Modulus of the material varied with respect to aging duration and is shown in Figure 6. The rate of degradation of modulus was dependent upon the rate of moisture uptake and fiber treatment process. At the end of the ageing period, TFRC and UFRC showed a reduction of 15% and 11% in modulus as compared to pristine samples.

Flexural Test

The flexural characteristics of UFRCs/TFRCs in pristine and aged conditions are studied at regular intervals as per ASTM standards and is shown in Figure 7.



From the results, it can be understood that UFRCs exhibit

Figure 6: Percentage reduction of Modulus in UFRCs and TFRCs



Figure 7: Variation of flexural properties

better flexural strength. Also, the flexural strength of pristine composites (both treated and untreated) was better than their aged counterpart. In the case of pristine specimens; UFRC exhibited a maximum flexural strength of 84.24±8.17 MPa while that of TFRCs was 64.10±4.068 MPa. At the end of the ageing process, UFRC and TFRC showed 17% and 22% reduction in flexural strength respectively.

Analysis of failure surfaces

To understand the effect of the ageing process on hemp/ epoxy laminates Scanning electron microscope (SEM) images of the tensile fracture specimens were captured and compared with pristine samples. Figure 8 (a) and (b) shows the SEM images of pristine UFRC and TFRC.

SEM images clearly showed the effect of fiber surface treatment on the wetting and coupling process between fiber and matrix interphase. Untreated fibers showed better bonding (Fig. 8(a)) between fiber and matrix surfaces. Inter fiber cracks along the loading direction are the major contributor to the failure of laminates. But in the case of TFRC poor bonding between fiber/matrix interphase is visible (Fig.8 (b)). Treatment of fibers with KOH appeared to have a smooth surface and may result in poor interlocking between fiber/matrix interphase and an overall reduction in tensile and flexural properties.

Figure 9 (a) and (b) show the tensile fracture surface UFRC and TFRC of aged specimens. Even though fiber treatment reduced moisture absorption rate as compared to untreated hemp/epoxy laminates; the poor bonding between fiber/matrix surfaces resulted in the decomposition of matrix material from the fiber surface. In the case of UFRC greater bonding observed and tensile loading induced cracks in the matrix surface and resulted in the formation of flakes of epoxy.

CONCLUSION

The present study was carried out to understand the effect of fiber treatment and the marine environment on the mechanical properties of Hemp/Epoxy laminates. Fibers





Figure 8: SEM images of tensile fracture specimens (a) UFRC, (b) TFRC



Figure 9: SEM images of tensile fracture specimens after 150 days of ageing (a) UFRC (b) TFRC

were treated with a 5% KOH solution. The tensile and flexural characteristics of specimens without treatment were slightly better than the treated counterpart. This is owing to increased alkaline concentration which leads to fibrillation of fibers also makes them stiff and reduced interlocking mechanism of fiber/matrix interphase. Treatment of fiber showed a slight difference in moisture absorption behavior and treated fiber-reinforced laminates absorbed lower moisture content. SEM images revealed the poor adhesion between matrix/fiber interphase in the case of treated fiber-reinforced hemp/epoxy laminates. Hence the treatment of fibers beyond a certain concentration of alkali solution will lead to poor mechanical characteristics.

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