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Research Article

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Effect of Mercerization on Mechanical Properties of Jute/Flax/Hemp Fiber Reinforced Polymer Composites

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Abstract

The primary goal of this study is to examine the impact of mercerization over elastic and strength properties of jute/flax/hemp fiber-reinforced polymer composites. Bast fibers such as flax, jute and hemp fibers were selected as a reinforcement in the composite, and epoxy was considered for the matrix. Using the Hand Lay-up method, the composites are manufactured with a 40% fiber volume fraction and a 60% matrix volume fraction. To enhance the fiber-matrix interface, all fibers are chemically treated by the process of mercerization. The natural fibers are treated with an alkaline solution (2% NaOH; sodium hydroxide). Tensile and compressive tests on composite coupons were carried out using the ASTM standards to investigate the material properties along various fiber directions of 0°, 90°, and 45°. Based on the results of mechanical properties, it was observed that the chemical treatment had mixed results over the composite properties. The mercerization had a positive impact on the composite's properties along the fiber direction, whereas the mercerization had a negative impact along the transverse direction of the fiber.

Keywords

Natural fibers, Mercerization, Sodium hydroxide, Mechanical properties, ASTM standards

Introduction

In the past few decades, the awareness about controlling the non-biodegradable waste from synthetic fibers has been increasing daily, and researchers are showing attention to biodegradable fibers to manufacture composites [1]. Natural fibers produced from plants and animals have satisfied this demand, especially bast-based fibers such as flax, hemp and jute. They have many benefits compared to synthetic fibers, such as low price, biodegradability, and minimal environmental influence [2]. The major drawbacks of natural fiber-reinforced polymer (NFRP) composites are mechanical characteristics compared to artificial fiber-based composites. Hybridization enhances the mechanical characteristics of the NFRP composites according to industrial needs. Numerous variables, including the choice of fiber and matrix, sequence and orientation of fiber, method of manufacturing, and fiber and matrix volume fractions, affect the properties of the composites [3].

The other main drawback of using bast fibers is their weak bonding with a matrix. Composite performance is influenced by the interfacial zone of reinforcing material and matrix. Generally, bast fibers are hydrophilic and the matrix is hydrophobic. Due to this diverse nature, fiber develops a poor bond with the matrix, which reduces performance of the composite. For this reason, the natural fibers are to be modified chemically to enhance the interface with the matrix. The literature shows various techniques to modify fibers chemically; those are mercerization, acetylation, benzoylation, acrylation, silanization, and carboxylic [4].

Among the different methods of chemical treatments, many researchers have considered the mercerization process. NaOH solution is considered in the mercerization process to alter the fiber's surface. The mercerization process develops the rough surface over fiber and increases the adhesion between the composites' reinforcing material and epoxy [5]. Many researchers have considered different concentrations of NaOH at various durations to enhance composite performance. When treatment is carried out with concentrations of 3%, 6%, and 9%, among these 6% treatment produces better results. As in the case of duration considered, a different study was conducted on the duration of 44 h and 148 h and identified that 44 h exhibited better mechanical properties [6].

In spite of the many benefits, there are some disadvantages to considering plant fibers as a reinforcement material for composite fabrication. Due to the lower strength of the NFRP composite, these are not useful for heavy-loaded structures. Natural fibers are prone to insect, fungal, and bacterial attacks and have low durability in an alkaline type of environment [7]. The chemical treatment during the manufacturing process mainly influences the durability of the natural fiber composites [8]. Treatment of fiber reduces the moisture absorption capacity of the fiber, and automatically it leads to the low weight of the composite. The objective of the current research work is to investigate the strength and elastic properties of the flax fiber reinforced polymer (FFRP) composite, hemp fiber-reinforced polymer (HFRP) composite, and jute fiber-reinforced polymer (JFRP). As the same, treated flax fiber reinforced polymer composite (TR-FFRP), treated hemp fiber reinforced polymer (TR-HFRP) and treated jute fiber reinforced polymer (TR-JFRP) composite mechanical properties were also determined. The mercerization process is used for the treatment of fibers.

Using the Hand Lay-up method, the composites are manufactured with a 40% fiber volume fraction and a 60% matrix volume fraction. Elastic properties such as modulus of elasticity (E), shear modulus (G), and strength properties like compressive, tensile and shear strength are determined according to the ASTM standards, i.e., ASTM D3039 [9] and ASTM D3410 [10]. For the material characterization, the loading orientation of material property testing is shown in figure 1.

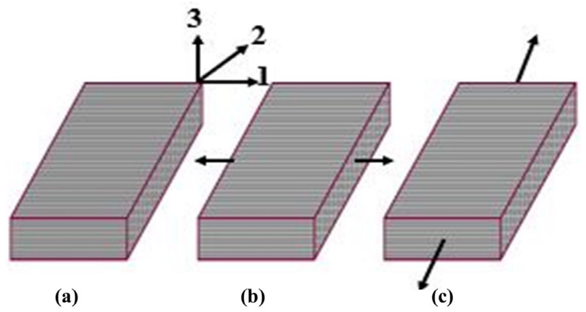

Figure 1: Loading orientation for material characterization of uni-directional composites **(a)** Unidirectional laminate, **(b)** Fiber direction, and **(c)** Transverse direction.

Methodology

Materials

Three types of natural fibers (Jute, flax, and hemp) are considered as reinforcement in the composite. The Fiber region, Chennai (India), supplies unidirectional mats of natural fibers. In this work, the matrix was prepared by the epoxy resin (Resin 691) and hardener (Reactive Polyamide of grade140) proportion of 90/10 by weight, respectively. The Shiva instruments & labware, Delhi (India) supplied resin and hardener. The considered fibers are treated with an alkaline solution (2% NaOH) to improve the surface roughness of the fibers. The pellets of NaOH were purchased by Varun industries, Pilani (India). For this research work, the fiber volume fraction of 40% was considered. Table 1 displays the mechanical characteristics of the fibers and resin.

Mercerization

The matrix-fiber interface in NFRP composite is a reaction zone that combines two phases chemically and mechanically. The matrix and fiber's adhesiveness influences the composite's mechanical properties. Among different treatment methods, the alkali treatment is one of the suitable methods to take off the lignin, hemicellulose, oil, and wax present around the fiber, enhancing cellulose amount and resulting in better mechanical properties [12]. The mercerization process helps to develop a rough surface on fiber when the fibers are treated with NaOH solution, which eventually improves the matrix-fiber adhesion property. Treatment of fibers with NaOH solution involves mainly four types, such as for a continuous period using constant NaOH concentration solution, for a continual period using different NaOH concentration, for different

Table 1: Mechanical characteristics of Jute, Flax, Hemp, and Resin [11].

Fiber type	Mechanical characteristics of resin and fibers					
	Density (ρ)	Tensile strength (P)	Young's modulus (E)	Specific modulus (E/ρ)	Elongation	Specific gravity (ν)
	(g/cm^3)	(MPa)	(GPa)	(GPa)	(%)	-
Flax	1.41	330 - 1550	30 - 82	16.6 - 55	1.2 - 3.2	1.52
Hemp	1.47	570 - 950	75	48	0.9 - 3.1	1.55
Jute	1.45	390 - 860	10 - 33	6.8 - 20.6	1.5 - 2.2	1.35 - 1.5
Resin	1.14	65 - 80	3.6	2.4 - 3.7	3.4 - 4.2	1.34

time periods treating with constant NaOH solution and for different time periods treating with different NaOH solution concentration [13]. Figure 2 shows the surface structure of the fiber before and after the mercerization process.

Processing of composite plate

The fibers are immersed in a 2% NaOH solution at room temperature for 1 h. The fibers are cleaned using distilled water to eliminate any NaOH residue on the fibers and kept in an oven to dry the fibers at a temperature of 40 °C for one day. The Hand Lay-up technique was used to fabricate composite by placing fibers and matrix in alternative layers in a steel mold. The whole assembly is kept in an oven for curing at a temperature of 80 °C for 3 h duration. Once the curing period was over, the samples were kept at room temperature till the time of testing the specimen. To evaluate the mechanical properties, samples are drawn out from the composite plate in the direction of 0°, 90°, and 45°, referring to the fiber direction. For each test, three samples are considered from laminate in every direction for tensile and compressive tests. The preparation and testing of specimens involve the steps of mercerization, composite plate manufacturing, cutting, and testing of specimens. The entire methodology is shown in figure 3.

Testing of composite specimens

ASTM D3039 was used to conduct tensile and compressive tests using ASTM D3410 standards. Table 2 describes specimen ID'S.

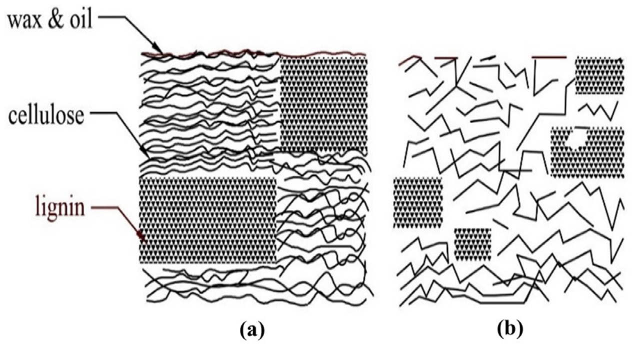

Figure 2: Surface of fiber **(a)** without treatment and **(b)** Treatment

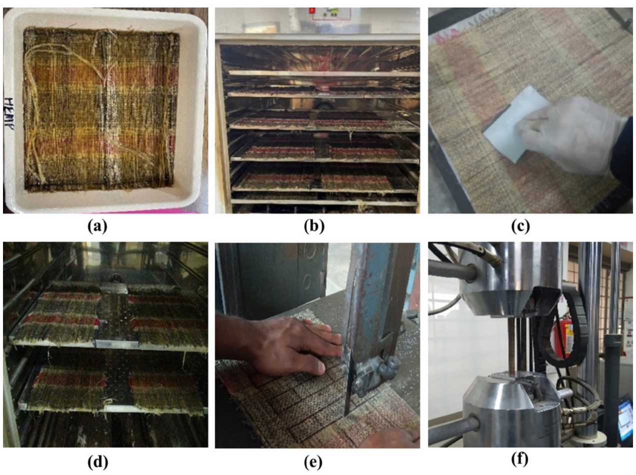

Figure 3: Preparation and testing of composite specimen: **(a)** Soaking of fibers in NaOH solution, **(b)** Drying the fiber at 40° C for one day, **(c)** Applying epoxy on fibers to manufacture composite, **(d)** Drying the laminate at 80° C for 3 h, **(e)** Cutting of specimens from laminate, and **(f)** Testing of composite coupon.

Table 2: Nomenclature of composites.

Composite type	Sample ID - tension test	Sample ID - compression
FFRP	T - FFFF	C - FFFF
TR-FFRP	T - TR-FFFF	C - TR-FFFF
HFRP	T - HHHH	C - HHHH
TR-HFRP	T - TR-HHHH	C - TR-HHHH
JFRP	T - JJJJ	C - JJJJJ
TR-JFRP	T - TR-JJJJ	C - TR-JJJJ

Note: 'T' represents tension, 'C' represents compression, and 'TR' represents the treated specimen.

Tensile and compressive tests

A universal testing machine of 100 kN capacity performed the tension and compression tests. The maintained crosshead speed in tensile testing is 2 mm/min, and the crosshead rate is 1.6 mm/min in the case of a compression test. The material properties like Young's modulus and strength of the composite in tension and compression are calculated experimentally to the fiber direction of 0° and 90°. The composite's shear modulus and in-plane shear strength are calculated when the fibers are tested along the 45° direction to the x-axis (longitudinal axis). The composite's shear modulus and in-plane shear strength are calculated using Eq (1) and Eq (2), respectively.

$$G_{12} = \frac{1}{\frac{4}{E_X} - \frac{1}{E_1} - \frac{4}{E_2} + \frac{2\nu_{12}}{E_1}} \tag{1}$$

Where, G_{12} is the modulus of the rigidity of the specimen, E_1 is Young's modulus of 0° direction; E_x is Young's modulus through the direction of loading; E_2 is Young's modulus of 90° direction; υ_{12} represents Poisson's ratio of specimen in the 1-2 plane. In-plane shear strength is also calculated using the stress transformation relations written for the 45° off-axis tensile test using Eq. (2).

$$\begin{pmatrix} \sigma_1 \\ \sigma_2 \\ S \end{pmatrix} = \begin{bmatrix} \cos^2 45 & \sin^2 45 & 2\sin 45\cos 45 \\ \sin^2 45 & \cos^2 45 & -2\sin 45\cos 45 \\ -\sin 45\cos 45 & \sin 45\cos 45 & \cos^2 45 - \sin^2 45 \end{bmatrix} \begin{pmatrix} \sigma_x \\ 0 \\ 0 \end{pmatrix} \tag{2}$$

Results and Discussion

This study aims to calculate the mechanical properties of NFRP composites, which include elastic modulus, the strength of the composite in tension and compression loading, shear modulus, and in-plane shear strength for treated and untreated FFRP, JFRP, and HFRP composites.

Tensile characteristics

The comparison of tensile strength of different types of composites and Young's modulus values are presented in the bar chart as shown in figure 4 and figure 5, respectively. Based on this figure, it is identified that the strength and modulus of chemically treated HFRP and JFRP composites along the fiber direction are higher than other composites. Mercerization negatively impacted the properties in the transverse direction.

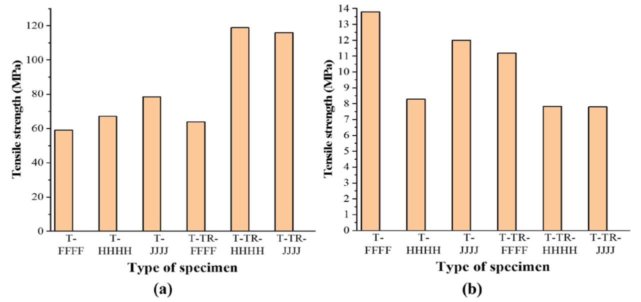

Figure 4: Tensile strength **(a)** Fiber direction and **(b)** Transverse direction.

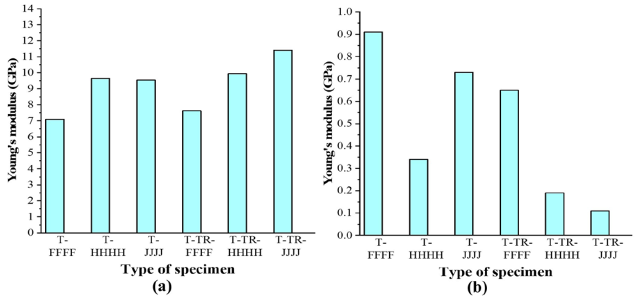

Figure 5: Young's modulus **(a)** Fiber direction and **(b)** Transverse direction.

The reason for this negative impact is that sometimes mercerization damages the fiber texture and creates fiber dispersion, which leads to a weak bond with the matrix. The FFRP composites have achieved better strength and modulus along the transverse direction than other composites.

Compressive characteristics

The comparison of compressive strength has been presented in the bar chart as shown in figure 6. Based on the figure, it was noted that the compressive strength of HFRP and JFRP composites have higher strength along fiber when compared to other composites. In the transverse direction, the FFRP composites produced better strength than other composites.

From the tensile and compression tests as per the ASTM standards, the elastic and strength properties of the different NFRP composites are calculated and presented in table 3.

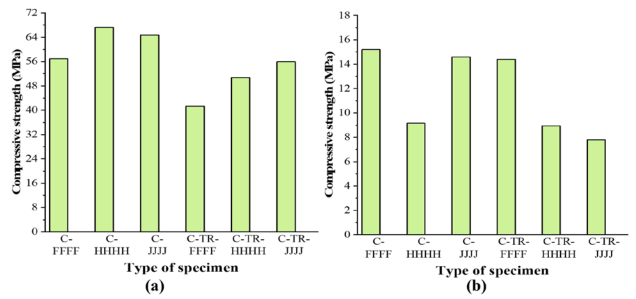

Figure 6: Compressive strength **(a)** Fiber direction and **(b)** Transverse direction.

Scanning electron microscope (SEM) analysis

After the tensile testing, morphological analysis was executed to identify the matrix-fiber interface and failure of the fiber composite. The samples with the maximum tensile strength in both cases were selected for this analysis. The treated and untreated fiber surface of the HFRP composite is examined using SEM. Before capturing the micrographs, the composite specimens underwent chromium coating to enhance conductivity. The micrographs of SEM for the HFRP composites developed in this study are shown in figure 7. The difference between the treated and untreated fiber can be observed in this figure by looking at the fiber surface. The untreated fiber has wax and oil around the fiber surface and in the treated fiber case, the fiber's surface is clean without any impurities. The fiber and matrix's interaction adhesiveness will

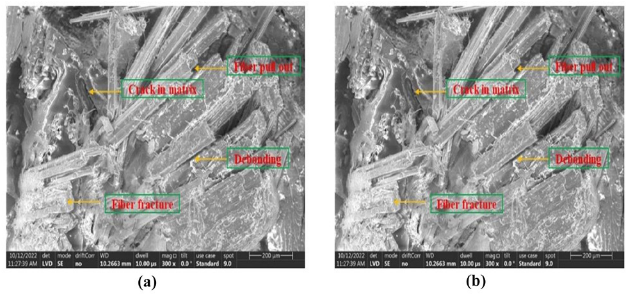

Figure 7: SEM images of HFRP composite after tensile testing along fiber direction **(a)** Untreated HFRP composite and **(b)** Treated HFRP composite.

Table 3: Material properties of the NFRP composite.

Laminate	E_1	E_2	G_{12}	X_t	X_c	Y_t	Y_c	S=T
	(GPa)	(GPa)	(GPa)	(MPa)	(MPa)	(MPa)	(MPa)	(MPa)
FFRP	7.09	0.91	0.24	59.2	56.9	13.8	15.20	9.41
TR-FFRP	7.63	0.65	0.27	63.9	41.3	11.2	14.4	5.74
HFRP	9.64	0.34	0.21	99.0	67.2	8.29	9.15	5.68
TR-HFRP	9.94	0.19	0.30	119.0	50.8	7.82	8.93	5.01
JFRP	9.55	0.73	0.19	78.5	64.8	12.00	14.60	5.92
TR-JFRP	11.4	0.11	0.22	116.0	56.0	7.79	7.80	3.03

Note: E_1 and E_2 = Young's modulus of elasticity along the longitudinal and transverse direction to fiber; G_{12} = shear modulus of plane 1-2; X_t, X_c, Y_t, and Y_c = tensile and compressive strengths of lamina along the longitudinal and transverse direction to fiber, respectively; T and S = shear strength in plane 1-2 and 1-3, respectively.

be influenced by the oil and wax content around the fiber. The SEM images also show a fiber and matrix crack, the fiber's pullout, and debonding.

Conclusions

This work examined the response of mercerization to the mechanical characteristics of various NFRP composites. Based on the results, the following conclusions are made:

- The mercerization process enhanced the strength and elastic properties of the NFRP composites along the fiber direction. Treated HFRP, JFRP, and FFRP composites had better strength in tension and modulus of elasticity than untreated HFRP, JFRP, and FFRP composites.
- The mercerization process negatively impacted the properties of the composite along the transverse direction. Untreated FFRP composites have produced better tensile strength and Young's modulus over other types of composites.
- Mercerization process bettered the shear modulus of treated composites and in the case of compressive strength, the mercerization had a negative impact.
- In the future, NFRP composites can potentially replace GFRP composites in applications that require low to medium load-bearing capabilities.

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Conflict of Interest

The authors declare no conflict of interests that are relevant to the content of this article.

Credit Author Statement

P. Siva Sankar: Methodology, Experimentation, Writing - original draft preparation; S.B. Singh: Resources, Writing - review and editing, Supervision. All the authors read and approved the manuscript.

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