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Hybridization of Chemically Treated Hemp and Pineapple Leaf Fibers in Thermoplastic Composites: Mechanical Performance and Moisture Resistance

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Researchers are showing a lot of interest in the possibility of using natural fibers as reinforcing agent in thermoplastic matrix to make cheap, lightweight composites. Despite the many benefits these fibers offer over synthetics, mechanical properties of composites include low wettability, excessive moisture absorption, and inadequate matrix-fiber adhesion are drawbacks. This experimental effort uses a hand lay-up process and chemical treatment on fibers to produce composites that blend two plant fibers originating from leaves, therefore avoiding these difficulties. The composites were exposed to an array of experimental trials. Hemp and pineapple leaf fiber (PALF) and Sodium hydroxide (NaOH) treatment together enhanced the composite's mechanical properties. Composites' compressive strengths are significantly greater than those of individual fibers, although their flexural values are marginally lower. Hydrophilicity is a common property of composites, and the results demonstrated that affinities for moisture content declined with age due to the effective chemical treatment on the fiber surfaces. The experimental data shows that the characteristics of the composites are significantly affected by the hybridization of hemp and PALF along with Sodium hydroxide (NaOH) treatment. The results of the investigation using scanning electron microscopy (SEM) of cracked image have been thoroughly reviewed.

Keywords: Hemp fibers; flexural strength; tensile strength; chemical treatment; mechanical properties; SEM

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An increasing number of people are concerned about environmental contamination, and fiber-reinforced polymer composites are in great demand due to their durability and other advantageous chemical and physical properties [1]. Artificial resin and synthetic fibers were revolutionary for decades due to their improved strength and stiffness, but they also contributed to environmental problems [2]. In an effort to address the serious issues with synthetic fibers, including their lack of renewability, health risks, and biodegradability, a number of academics have been working on finding alternatives. With the growing interest in natural fibers, a viable substitute for synthetic reinforcing should be available soon [3]. These fibers are naturally produced by a wide variety of animals and plants, including rabbits, silk moths, camels, hemp, pineapples, bananas, coir, palm trees, flax, jute, and countless more. Composites manufactured from natural fibers derived from plants have several advantages, including being cheap, biodegradable, and easily accessible [4]. Nevertheless, the material's qualities, including tensile strength, stiffness, chemical resistance, and heat resistance, will be significantly improved due to the combined nature of fibers and fillers [5]. Because plant fibers are so absorbent of moisture and don't mix well with other materials, scientists have had to resort to hybridization with either conventional or filler fibers. The individual components will always be able to hold their own, due to the cohesiveness of the whole [6]. The combination of different materials might lead to composites with unwanted properties. For example, a material that was reinforced with peanut oil cake and comprised of flax and pineapple fibers had enhanced tensile, flexural, impact, and bending strengths. Composites made with silica improve their mechanical performance, whereas those made using hybrid kenaf/pineapple fibers and filler are lighter [7]. The flexural strength (FS) was rised by 25.46 % and the tensile strength (TS) by 18 % using alkali-treated PALF with 3% powders. This was due to the PALF's outstanding adhesive properties and improved high density deposition rate [8]. The aggregates will disappear after ultrasonicating the solution for a long time. The shape of the reinforcement's failure surface changes during hand lay-up, in contrast to the glassy and multi-crack behavior of pure epoxy [9]. The combination of these properties increases the hardness, flexural strength, tensile strength, and compressive strength by 33.0 %. Yet, the material's binding strength was decreased and brittleness increased to 10 % particle loading. Composites enhanced with PALF particles are more biodegradable due to their enhanced thermal stability and water absorption. The TS, FS, and FM values for the PALF/viscose mixture

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are 21.8 MPa, 24.6 MPa, and 718.8 MPa, respectively, when it comes to bending [10]. Since viscose is the main component causing a decline in TS, including PALF has improved it. The uneven distribution of the epoxy resin also caused a drop in Young's modulus. Due to its anti-moist absorption, uniform distribution, and strong mechanical characteristics, PALF/viscose yarn is used to make a wide variety of products, including shopping bags, attractive roof coverings, coffee cups, and cans, and more [11]. The PALF showed remarkable properties when reinforced with epoxy resin. In comparison, PALF's tensile strength is 61.2 % greater and its tensile modulus is 49.5 % higher. The low micro-fibrillar inclination allows for maximum fiber/matrix attachment, and the high cellulose content (82 %), which causes a rougher surface [12]. Consequently, materials find their way into building components, electrical packages, and automobiles. As a result of its strong binding ability, PALF outperformed other plant fibers in terms of anti-slip characteristics and reduced specific wear rate. The main cause of the composites' properties is the processing-incorporated 40 wt% TiO_2 filler. Hence, PALF provided the best circumstances for wear and frictional applications, and low wear characteristics ranging from 20 to 40 wt.% were achieved using the Taguchi technique [13]. The roselle/PALF hybrid fiber resulted in a 500m wear rate reduction at a sliding distance of 1m/s. Applying a 5 N load during testing at this sliding speed significantly improved the tensile, flexural, and impact characteristics. This has a major impact on the tribological properties of composites reinforced with hybrid fibers [14]. In this study, a hybrid composite was created by hand-laying up reinforcements made of coconut shell powder. Our goal in performing these investigations and evaluations was to gain a better understanding of the manufactured composite materials' characteristics and strength. Since the experimental results demonstrate that the combination of the two fibers significantly alters the composites' characteristics, hemp and PALF fibers might be the innovative fibers for polymeric based composites.

EXPERIMENTAL METHODOLOGY

Materials

The composite's epoxy resin (LY556) and hardener (HY951) were provided by Kovai cheenu enterprises of Coimbatore, Tamil Nadu, India. In order to enhance the composite, Fiber source firms in Chennai, TN, India, provided PALF and hemp fibers. The gathered fibers were sized, rinsed triple times with water, and then dehydrated in direct sunshine in an airtight container. The surface texture of the fibers was then improved by treating them with a NaOH solution for three hours then the fibers from the NaOH solution, rinsed them with deionized water, and allowed them to dry for up to four hours [15]. The experimental study utilized a stoichiometric ratio of 10:1 for the addition of the matrix and hardener.

Fabrication

The hybridized composites were prepared using the manual layup process. The correct quantities (10:1) of epoxy resin and hardener were added to a beaker, and the mixture was meticulously stirring for four minutes at ambient temperature using a magnetic stirrer. Then, for the purpose of precision, it was left to stand for 60 seconds [16]. The end product was a mixture that was only partly solid. After just one minute in liquid form, the hardener and resin's adhesive force was sufficient to dissolve the molecular bonds. The mold was protected by covering its surface with wax substance and placing a polythene sheet on its underside. At room temperature, the fibers (30 wt. %) were incorporated into the liquid to create a composite. This matrix and hardener only require room temperature to cure. Fig. 1 shows the composite processing flow, beginning with the mold and ending with the produced laminate.

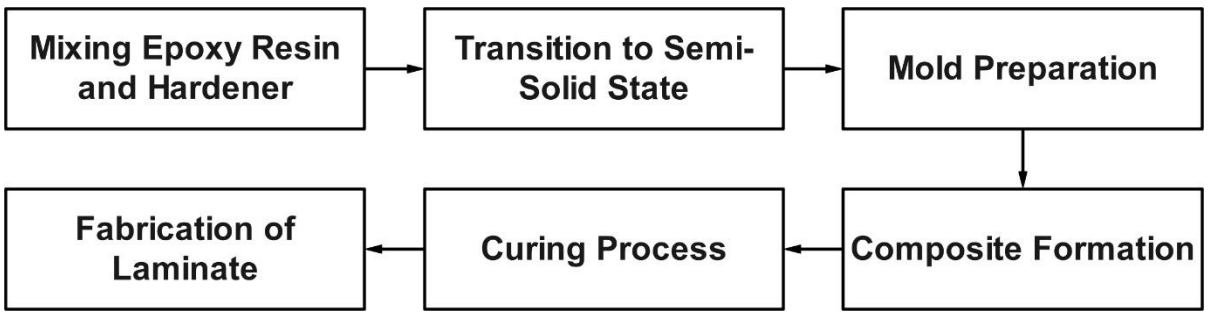

Figure 1. Fabrication of composites.

EXPERIMENTAL ANALYSES

Analysis of Mechanical Properties

Mechanical testing, including tensile, compression, flexural, and impact tests, was used to study the influence of fiber loading on hybrid composites. The experimental inquiry involved the fabrication of hybrid composites using the hand layup technique to reinforce PALF and hemp fibers. The specimens were tested for tensile, flexural, compressive, and impact strengths according to the standards set by ASTM.

Tensile Properties

The TS is enhanced because the matrix is exchanged with the fiber in a periodic and regular manner. The composite's TS is enhanced due to the improved adhesion between the matrix and fibers. The interface's TS drops as a result of van der Waals forces' weak bonding, which weakens the matrix and fiber. To perform the test, a UTM with a crosshead speediness of 1.5 mm/min was utilized. As outlined in ASTM D3039, tensile testing techniques. Among the most crucial and extensively studied material and structural application qualities is its resistance to breaking under tensile stress.

Flexural Test

To resist deformation under stress, a material must have a high flexural strength, which is also called its modulus of elasticity or fracture strength. Following the guidelines laid out by ASTM D638 for flexural testing, the identical UTM was used. The following specifications are followed during the test: The crosshead speediness of 1.5 mm/min, and the supporting length is 100 mm. The trials were conducted using a 3- bending flexural test.

Compressive Strength

Composites were subjected to this test in order to determine their average compressive strength. The compressive strength of epoxy strengthened with hemp fibers and a PALF hybrid was examined in a controlled laboratory setting. The prepared specimen was subjected to axial compression loading using the same UTM. A process defined by ASTM D695 was used in the production of the specimens. The sample is placed in the test fixture, which is then loaded in compression and placed between the testing machine's plates, in order to achieve the compression test result. The maximum load that was achieved after the sample breakage was recorded by the UTM machine.

Impact Strength

Impact strength sample were manufactured with dimensions of 65 mm × 13 mm × 3.2 mm with a V-shape notch as per ASTM D256. The findings of the specimens' impact tests were documented after they were run through the Tinius Olsen Impact 104 machine. For every scenario, we examine three samples and utilize the average data for analysis.

Water Absorption Studies

During the initial treatment, we measured the amount of water absorbed using a moisture absorption test. A 24-hour immersion in room-temperature water is the following phase. Placing the composite samples in a tray filled with distilled water allowed us to estimate the water absorption percentage, while Eq. 1 was used to determine the weight gain percentage. We documented the sample dimensions according ASTM D570's specifications.

$$\% \text{ Weight gain } = \frac{m_f - m_i}{m_i} \times 100 \quad (1)$$

where the starting weight of the samples is m_i and the ending weight is m_f.

X-Ray Diffraction (XRD) Analysis

The XRD method was used to examine the physical properties of the composites made from PALF and hemp fibers. The experiment used scanning values of 2θ ranging from 10º to 80º to acquire X-ray diffractograms under the conditions where CuK (wavelength 1.54Å) 2θ 5s-1. Equation 2 was used to compute the crystallinity index (CI), while equation 3 is supplied by Scherrer as the formula for calculating the crystallite size [17].

$$\text{CI} = (\theta_{22.55} - \theta_{18.5})/\theta_{22.55} \quad (2)$$

$$\text{CS} = \frac{K\lambda}{\beta \cos\theta} \quad (3)$$

In this case, θ stands for Bragg's angle, β for the peak width at half maximum, K=0.89 for Scherrer's constant, and λ for the radiation wavelength.

Fourier-Transform Infrared Spectroscopy (FTIR) Analysis

The functional group of a chemical applied to the composite surface was investigated in this work using FTIR. Using a Perkin Elmer Spectrum, functional groups were investigated in the 400 to 4,000 cm^{-1} infrared region in this experimental work [18].

SEM Analysis

The microstructure of the PALF and hemp fiber strengthened hybridized composite's fractured surfaces was investigated using a scanning electron microscope, among other tools. We also examined the efficiency with which the cellulose fibers absorbed the energy provided during loading. The samples were scanned at the Bannari Amman Institute of Technology using a Zeiss scanning electron microscope.

RESULTS AND DISCUSSIONS

Results on Mechanical Behavior

The data collected from the mechanical testing experiments are presented in Table 1. Three samples were evaluated at room temperature to determine the final composite average values. Composites made from PALF and hemp fibers and mechanically loaded to compare their strengths are shown in Figure 2(a) to Figure 2(c). The findings highlighted the importance of hybridization in defining the composites' strength.

Tensile Properties

Figure 2(a) displays the findings of the tensile properties. Results showed that tensile strength values varied between samples, with differences seen according to elongation and cross-sectional area. The cellulose content and fiber bundling features of PALF and hemp fiber-reinforced hybrid composites are responsible for their maximum tensile strengths of 2.807, 3.849, and 4.962 kN, respectively [19]. The tensile strength is lower in PALF-reinforced composites related to other types of composites due to the voids in the material. Cracks and fiber pullout are the two most important factors in determining the reaction to tensile failure [20]. In addition to having a diameter of 0.3 to 0.4 mm, PALF has a strength of about 80 N. An average tensile strength of 1,086 N· mm−2, an elongation of 19.8 %, and mechanical characteristics ranging from 10-40 GPa were achieved with hemp fiber.

Flexural Properties

Figure 2(b) displays the outcomes of a flexural strengths of various composites to determine their bending capacity. The figure clearly shows that the flexural strength of the composites is enhanced by raising the fiber adding with the polymer. The flexural strength decreases as the loading of fibers increases because of their interaction. Findings indicate that macromolecule mobility within the epoxy and fiber improves FS [21]. This will prevent the composite from beginning to break. It would appear that the enhanced diffusion rate between the matrix and PALF or hemp fibers allows them to withstand significant weights. Hemp fibers were twisted at their origin, which reduced the composite's flexural strength [22].

Compressive Strength (CS)

Figure 2(c) displays the outcomes of a CS of various composites. Once the composite is no longer subjected to stresses, its restoration ability can be determined. Compression loading resistance of the composite is also calculated. The findings show that the hybridized composites have a maximal CS of 3.927 kN, whereas the PALF and hemp fiber composites have ultimate compressive strengths of 2.634 kN and 2.116 kN, respectively. All of the composite samples that were evaluated showed significant differences in compression loading. The results show that these hybrid composites can resist high loads better than other plant fiber composites. This is because the composite is more resistant to transverse delamination caused by external forces, which slows down its tendency to fail [23].

Table 1. Mechanical characteristics of the fibers.

Composite sample	Peak Tensile Load (kN)	Peak Flexural Load (kN)	Impact strength (N/mm^2)	Peak Compressive Load (kN)
PALF	1.46	1.86	44	2.37
Hemp	2.49	1.52	43.5	1.83
Hybrid	3.59	1.65	43	3.63

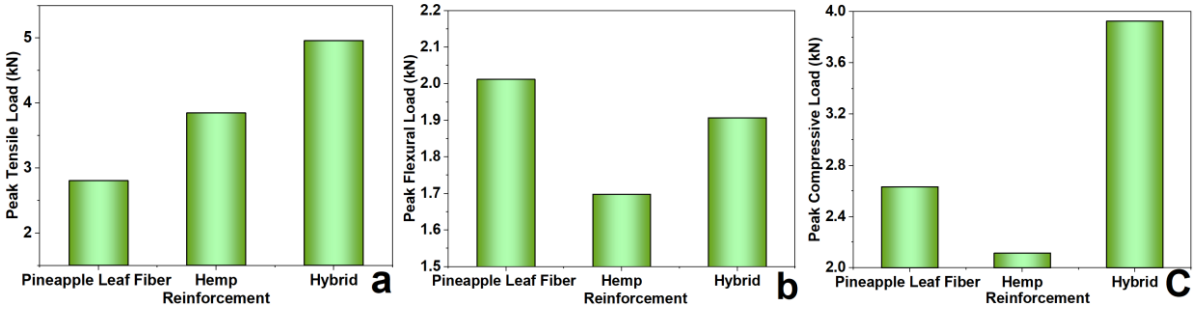

Figure 2. Evaluation of peak (a) Pensile load (b) Flexural load (c) Compressive load of the fiber reinforcement.

Impact Strength

Composites reinforced with PALF and hemp fibers separately have much greater impact strengths than the hybrid composite. The strength of PALF composites is surpassed by those reinforced with hemp fibers. For this experiment, the fibers are strengthened by being oriented longitudinally. As shown by SEM images, the weak interfacial connection between the matrix and the fiber is the primary cause of the meager impact strength [24]. The hybrid PALF/hemp composite shows no discernible alterations.

Water Absorption

The initial weight was employed to calculate the amount of water that the samples absorbed, which in turn determines how much their weight increased. The sample is able to absorb a greater quantity of water due to its porosity. The fiber has an appropriate porosity level, so it can absorb more water, but the sample has a lesser pore capacity, so it can only contain so much water [25].

As a proportion of the individual sample's weight rise over time and year, with no change in temperature, Table 2 depicts the water absorption property of the composites. The absorption rate increased continuously until saturation after immersing all samples in water. The weight of the composites increases after 24 and 48 hours at 28.3ºC and 28ºC, respectively, as shown in Figure 3a. When compared to epoxy resin, PALF's improved absorption property is indicated by the percentage increase in weight. Researchers have shown that epoxy with a year's worth of age has a better absorption property than freshly purchased epoxy. The absorption rate is higher when epoxy resin is held on hydrophilic fibers [25], such as PALF and hemp (Figure 3b). This causes the composite to develop microcracks, which in turn reduce the resin's brittleness. As a result of micro-cracking, the matrix fails due to the fiber's enlargement. The capillary effect uses it as a conduit for the simple absorption of water molecules [26].

Table 2. Results on water absorption of the various fiber reinforcement.

S. No.	Specimen	Increase in mass for newly mixed epoxy composites (g)		Increase in mass for 12 months old mixed epoxy composites (g)	
	Temperature of water	24 h @ 28°C	48 h @ 28°C	24 h @ 28°C	48 h @ 28°C
1.	PALF	2.06	1.96	4.83	4.83
2.	Hemp	2.27	6.09	5.02	2.76
3.	Hybrid	1.98	2.84	4.07	3.97

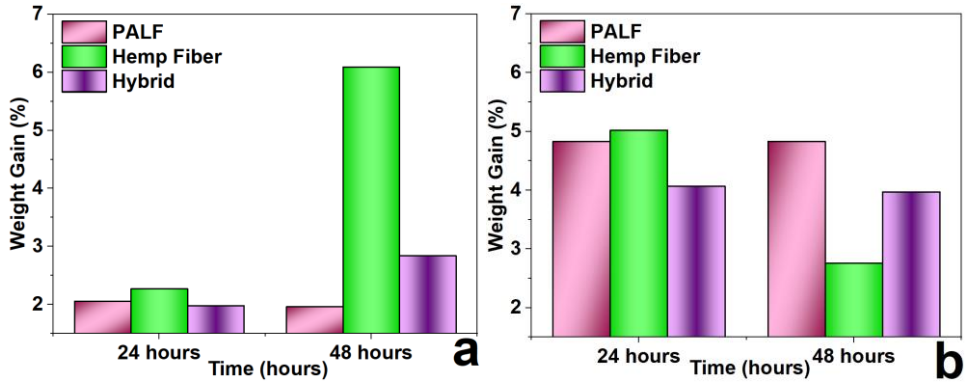

Figure 3. Evaluation of water absorption of (a) new composites and (b) 12 months old epoxy composites.

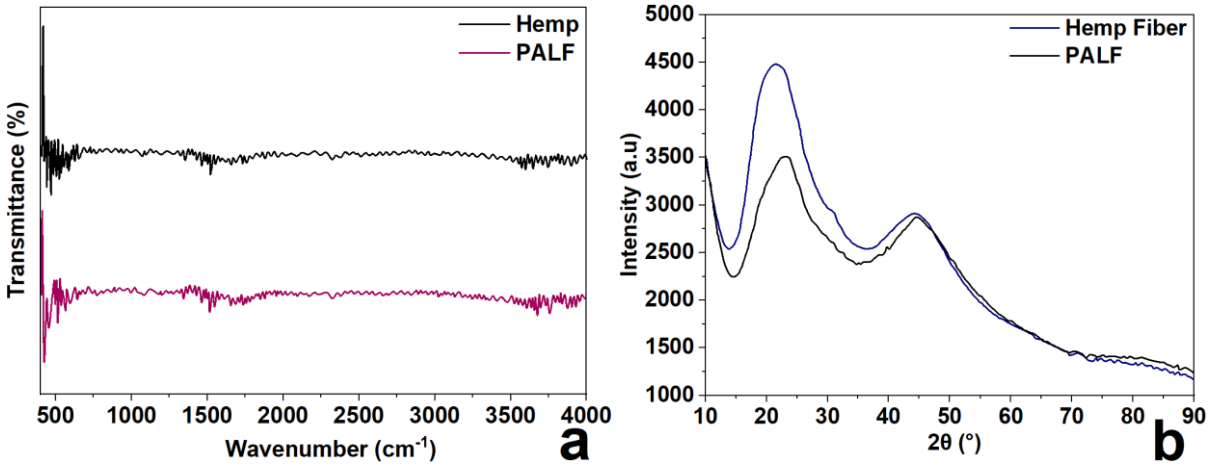

Figure 4. (a) FTIR pattern of Hemp fiber and PALF (b) Results on XRD analysis of different fiber composites.

FTIR Analysis

Figure 4 illustrates the spectral differences between hemp fiber and PALF composites. The strong hydroxyl group absorption [27] at 3,485 cm^{-1} confirms the O-H stretching vibration in cellulose fibers, while a weaker peak at 2,920 cm^{-1} corresponds to C-H stretching. A notable absorption at 1,653 cm^{-1} results from C-H bending vibrations. The alkalization process removes non-cellulosic components, leaving crystalline cellulose [28] with characteristic peaks at 1,423, 1,136, and 891 cm^{-1}. PALF composites reinforce hydroxyl absorption at 3,444 cm^{-1}, while hemicellulose, initially observed at 1,695 cm^{-1}, diminishes due to C=O stretching. NaOH treatment effectively removes lignin, wax, and oil, identified [29] at 1,464 cm^{-1} and 2,900 cm^{-1}, respectively. FTIR analysis of PALF composites also reveals hemicellulose and lignin at 1,761 cm^{-1}. Cellulose molecules exhibit flexing vibrations at 1,065 cm^{-1}, while C-O and C-H bonds contribute to additional stretching at 1,113, 1,388, and 1,413 cm^{-1}. The occurrence of a hydroxyl radical at 3,452 cm^{-1} in hemp fiber composites validates stretching vibrations and C=O bands. Tetrahedral absorption structures appear between 1,510-1,632 cm^{-1}, while C-O bond fragmentation is identified at 1,251 cm^{-1}. Peaks in the 552-590 cm^{-1} range suggest apatite formation on the composite surface.

XRD Analysis

By analyzing the composites using XRD, the crystalline phase may be located. In the 2θ range, which extends from 10° to 50°, the composite is scanned. The diffraction peaks are found at various angles within the defined range. Fig. 4 shows the peak values for the rutile phase at 31.18°, 38.82°, 40.53°, 49.06°, and 51.74°, which denote amorphous particles that depend on the crystalline area. These peak values are concordant with the statistics, particularly 89 to 1181 and 85 to 1290. Alkaline solution treatment of the fiber surface increases the crystalline diffraction level, leading to a rise in the amorphous level. The alkaline treatment has reduced the noncrystalline components, increasing the crystalline property [30], as seen by the diffraction peaks at 14.6°, 16.3°, 20.8°, and 22.9°. There is additional evidence that the mechanical characteristics of the composite are improved and the hydrophilic qualities of the fibers are reduced as the crystalline size increases. At 18.85°, 18.822°, 22.27°, 25.52°, and 32.47°, several large peaks were observed in the diffraction pattern. A substantial apatite layer development has occurred across the surface, suggesting that the composite layers are both robust and sharp. As a result of the alkaline treatment, which decreases the fiber's amorphous cellulose, the CI rise with time [31]. Analyzing the peaks revealed a diffuse pattern with distinct visible peaks, with peak overlap as measured by the Segal method causing the intensity between them to vary.

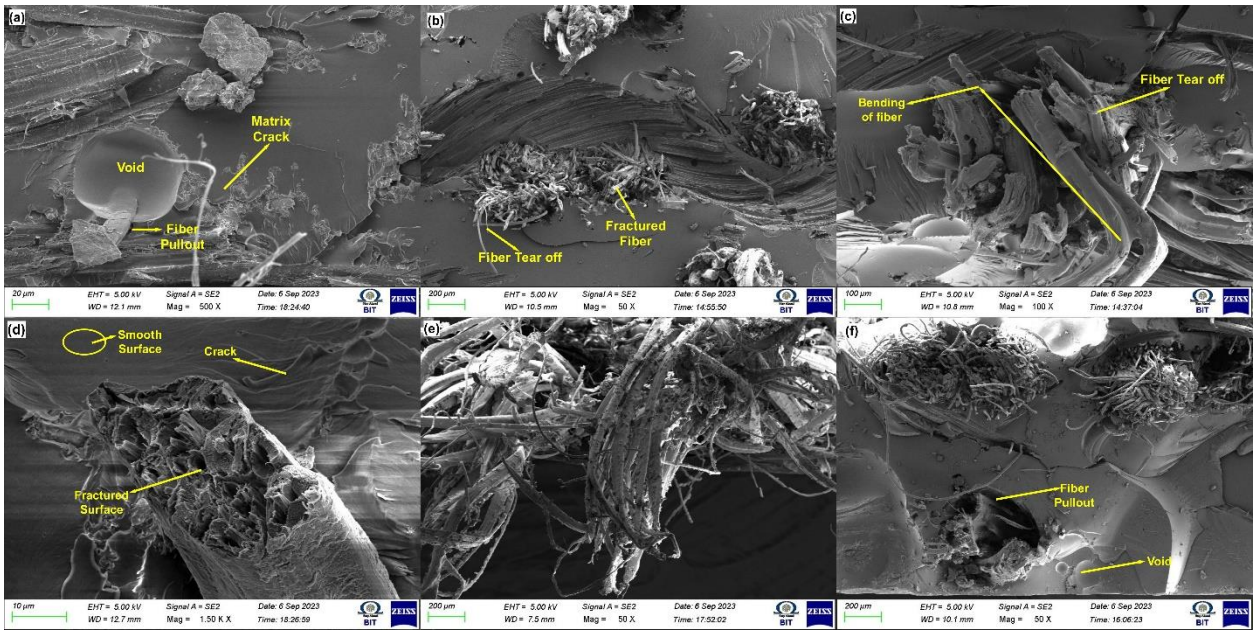

Figure 5. Scanning electron microscopic images of fractured surfaces of tensile loading, compressive loading, flexural loading of PALF composite (a-c) and hemp fiber composite (d-f).

SEM Analysis

Composite materials were analyzed for their morphological behavior in respect to scanning electron microscopy (SEM) pictures. Fig. 5(a) to Fig. 5(f) shows that tensile and flexural loading caused the PALF composite's shattered surface. Additionally, the alkaline treatment confirmed the decrease in hemicellulose. The composite has a smooth surface with visible pores. Fiber aggregation, fiber pull-out, parallel fiber cracks, and fiber tear-off broken fiber, are other findings revealed by scanning electron microscopy images. The cellulose molecule is negatively affected by the failure and fracture of the hemp fiber composite sample under mechanical loading [32], as seen in Fig. 5d to Fig. 5(f). The huge effect of the cellulose molecule on energy absorption causes it to occur eventually. Inadequate stress distribution impacts particle agglomeration and, in certain cases, causes failure. This demonstrates that cellulose pull-out, which may be lower or higher depending on the cellulose concentration, is a sign of composite failure [33]. There are signs of fiber pull-out on the surface of the hemp fiber composite, which is consistent with the fractured surface. Because agglomeration prevents adequate bonding at the fiber-matrix contact, it weakens mechanical strengths. There is strong evidence that the weak intermolecular attraction force causes the fiber and matrix to connect poorly, which in turn causes textural breakage [34].

CONCLUSIONS

Comparing the properties and behaviors of hand-lay-up PALF, hemp, and hybrid composites was the driving force behind this experimental study. When compared to the mechanical properties of hemp fiber and PALF composites when used separately, the hybrid's tensile strength of 3.59 kN is the superior choice. The flexural strength of the PALF/hemp hybrid fiber-reinforced composite is 1.65 kN, which is located in the middle of the spectrum between PALF's 1.86 kN and hemp's 1.52 kN figures. In a combination of hemp fiber and PALF, the total FS falls to 0.13 kN, which is the absolute minimum that may be maintained without flexurally weakening the structure. Crush strength of the hybrids is much greater than that of the other two fiber-reinforced composites. Because the matrix and fiber do not form a strong bond at the interface, hybrid composites have a low impact strength. The affinity toward moisture content was diminished as a result of the improvement in mechanical quality. Composites become rougher and have a larger surface area after being treated with an alkaline solution. Although the difference is small, this study shows that epoxy composites reinforced with PALF have a better water absorption property than those reinforced with hemp fiber, though the gap is not completely closed. Because hemp has a lesser concentration of cellulose molecules, its water absorption property is lower than that of PALF, which has a larger concentration of cellulose molecules. According to the FTIR measurements, the hydroxyl group that comes out of the PALF surface is responsible for the noticeable absorption peak at 3,521 cm^{-1}. The presence of tetrahedral structures, achieved through absorption on the hemp fiber's surface, is further supported by the peak range of 1,612 cm^{-1}. It is evident that the fillers are formless and reliant on the crystal-like region based on the XRD peak values at 29.97°, 36.43°, 39.82°, 47.97°, and 49.04°, which symbolize the rutile phase. The surface morphology of hemp fibers and PALF is quite similar and has not been altered significantly, according to scanning electron microscopy

(SEM) images. The mechanical strength decreases, for instance, when the intermolecular interaction at the fiber-matrix interface is insufficient.

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