Comprehensive Analysis of Physical, Mechanical, Morphological, and Water Absorption Properties of Bio-Polymer Composites Reinforced with Agricultural Waste Fibers

Vinoth Viswanathan

Department of Automobile Engineering, Easwari Engineering College (Autonomous), Chennai, Tamilnadu, India – 600089. Email: vinoth.v@eec.srmmp.edu.in

Sathiyamurthy Subbarayan*

*Department of Automobile Engineering, Easwari Engineering College (Autonomous), Chennai, Tamilnadu, India – 600089. Email: sathiyamurthy.s@eec.srmrmp.edu.in

Ananthi Narayanasamy

Department of Information Technology, Easwari Engineering College (Autonomous), Chennai, Tamilnadu, India – 600089. Email: nandhura@gmail.com

Ramalingam Senthil Kumar

Easwari Engineering College (Autonomous), Chennai, Tamilnadu, India – 600089. Email: rs_kmr@yahoo.com

ABSTRACT

Paddy straw fiber an agricultural by-product is frequently managed through incineration, representing a prevalent disposal method. There is a need for a solution to help reduce the harmful effects (air pollution, environmental issues) of burning rice straw. Efforts are being made to find sustainable and beneficial uses for paddy straw to reduce its environmental impact. The research gap is exploring the technologies for converting agricultural waste into innovative products. This paper describes the preparation of composites using paddy straw fiber-reinforced polyester bio-composites. The chemical treatment involves the paddy straw fiber to improve the fiber's strength. To delineate the inherent and modified fibers, a range of assessments were performed, encompassing single-fiber analysis, X-ray diffraction, Fourier Transform Infrared Radiation, Thermo gravimetric Analysis, and surface imaging. The strengths of raw and treated fibers achieved the values of 10900 gf and 8540 gf respectively. Randomly oriented composite laminates with the dimensions of 300x300x3 mm and different weight proportions of 70:30, 60:40, and 50:50 (Untreated and treated) were fabricated using semi-automatic compression molding. After fabricating the laminates, cutting the specimens using the ASTM standard for evaluation of the mechanical behaviors (Tensile, flexural, impact, hardness, and shear) and also studied the water absorption in each laminates, and preparation of the specimens to identify the morphology and structure using a scanning electron microscope. ALT-3 laminate achieved the maximum values of tensile, flexural (bend), impact, hardness, and shear values such as 20 MPa, 31 MPa, 62 KJ/m², 34 HV, and 39 MPa respectively. Overall results revealed that paddy straw fiber is a very superior material for nonstructural applications.

KEY WORDS: Paddy straw, Fiber Characterization, Mechanical properties, Morphology, Water absorption rate

INTRODUCTION

The increasing popularity of natural fiber-reinforced composites in polymer matrices is attributed to their myriad advantages across a diverse array of engineering applications.

Stubble waste management is the main goal of the research, replacing wooden-based products with agricultural waste. The products will be tested for replacing traditional table tops, wooden boards, particle boards, medium density fiber, and plywood. Natural fibers, deriving from distinct sources, can be categorized into three primary types: animal fiber, mineral fiber, and plant fiber. Plant fibers have shown excellent studyingperformance compared to other sources of fibers [1-4]. Natural fiber has incredible properties such as inexhaustibility, lower density, low cost, eco friendliness, non-toxicity, easily disposable, easy processing, handling, and degradability [5-7]. The primary attributes of fiber-reinforced plastics hinge upon various factors, including the nature of reinforcement (long fiber, short fiber, and particulates) [8-10], type of matrix (thermoplastic and thermosetting) [11-13], physical property (diameter, density, single fiber strength, crystallinity index) [14-16], fiber orientation (Angle wise, unidirectional, bidirectional and tri axial and randomly oriented) [17-19], chemical things (cellulose, hemi cellulose, lignin, wax etc.,) [20-22], amount of fiber loading in volume or weight percentage [23-25], layer sequencing and composite fabrication methods (handlay up, resin transfer moulding, and compression moulding) [26-29]. Ashok et al examined the mechanical, morphological and ballistic impact behaviour of luffa fiber strengthened different type of nano fillers with epoxy composites [30-32]. Ramesh et al studied the calotropis gigantea fiber reinforced thermos setting composites. Different fiber orientation was used in this study (0° to 90°). The mechanical properties achieved the maximum value for 0° orientations and also the composite can survive temperatures of more than 600°C, according to thermo gravimetric analysis and differential Scanning Calorimetry [33-34].

Muthu Choza rajan et al the chloris barbata flower fiber/epoxy composites were characterized through various analytical techniques following the alkali treatment of the fiber. Thermo Gravimetric Analysis (TGA), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Analysis (FTIR), and Atomic Force Microscope (AFM) were employed for comprehensive а assessment of the composites [35]. Ismail et al. this study delved into the mechanical characteristics of composite materials comprising a polymer matrix reinforced with rice straw fibers. Derived from agricultural residues, the rice straw fibers underwent a purification process and were then fragmented into small segments using a shatter machine crafted locally, prior to their incorporation into the investigation. After being ground, the fibers were separated by size using a sieve; their diameters ranged from 1.25 to 0.85 mm and their lengths from 20 to 8 mm [36]. Pan Ming Zhu investigated the crystallization behavior of the paddy straw fiber reinforced high density polyethylene composites. Paddy straw strand and refined fiber both exhibited higher aspect ratios of 16.31 and 14.48, respectively [37]. Ngo Dinh et al. examined that indications point towards a decline in the thermal stability of Polypropylene as a consequence of the inherently low thermal traits of Cellulose fibers. This reduction is particularly notable when Cellulose fibers are loaded to elevated levels. Nevertheless, it is noteworthy that the temperature corresponding to a 10% weight loss surpasses 300°C [38]. Samir Kamel et al reported that composites manufactured from processed rice straw with sodium hydroxide at 80°C produced the highest bending and tensile strengths. Composites treated with a coupling agent have better dimensional stability and mechanical characteristics than untreated ones. As lignin concentration rose, these qualities become stronger [39]. Akineymi et al the composite boards' waterproof water absorption and thickness swelling characteristics were superior to those of wood particleboard [40]. These affordable and easily customizable boards can be utilised to prevent impact damage. They can be used in place of flexible materials like insulating boards in construction. Numerous workers have reported on more research on wood-based fillers [53,54,55].

The deforestation and unsustainable logging practices lead to negative environmental effects which can be reduced by replacement of agricultural waste [44,45].

In the past few years, numerous researchers have been concentrated on agricultural wastages such as bagasse, wheat straw, paddy straw, etc., Paddy straw burning: a problem for the environment, agriculture, and humans, such as air pollution, breathing problems, traffic issues, eye problems, and so on. The stubble can be utilised in a variety of ways other than burning, such as animal feed, compost manure, roofing material for rural regions, a source of biomass energy, a growing medium for mushrooms, packing material, fuel, paper, bioethanol, and products for industry. Farmers and rural communities should be aware of how important waste management is for a sustainable future, instead of spending money on waste management, change of agricultural waste to value-added products and fabrication of eco-friendly products from the waste material will help generate wealth from the waste.

The novelty is efficient and sustainable utilization of paddy straw that is crucial for reducing agricultural waste, enhancing resource efficiency, and promoting environmental sustainability.

In this article studies were conducted on the fiber characterization, weight percentage of fiber loading (30%, 40%, 50%), alkali treatment of 5% sodium hydroxide, 24 hours of soaking time, mechanical behaviours (Tensile, flexural, impact, hardness, shear), water absorption, and morphological properties of paddy straw fiber reinforced polyester composites.

EXPERIMENTAL SECTION

Materials

Paddy straw, also recognized as rice straw, emerges as a by-product in the aftermath of rice harvests, encompassing the plant's stalks, leaves, and husks. This agricultural residue holds particular significance in regions where rice cultivation prevails. The unsaturated ortholamination polyester synthetic resins, denoted as VBR 2303, result from the chemical reaction between dibasic organic acids and polyhydric alcohols. To instigate the curing process, a catalyst (VBR 1204 - methyl ethyl ketone peroxide) and an accelerator (VBR 1201 - cobalt octoate) prove indispensable. Sourced from Vasavibala Resins Pvt. Ltd. in Chennai, the polyester resin, inclusive of the accelerator and catalyst, boasts properties such as a density of 1.132 g/cm³, a viscosity of 470 (cp) at 25°C, and a volatile content of 36.2% (wt).

Extraction of the Paddy straw fiber

The agricultural land is obtained after harvesting the many wastes. Hand-harvested paddy straw fibers collected on agriculture land at Thiruvallur (Dt) in the grade of manakaththai After collecting the paddy straw and remove the unnecessary fiber parts, the paddy stem is obtained. Fig. 1 shows the photographic image of the removal of paddy straw fiber.



Fig. 1: Photographic image of collecting the paddy straw fibers a. Agriculture field b. Paddy straw c. Paddy straw stem

Alkali treatment of the extracted fiber

The limited strength of raw paddy straw fiber is only due to some surface imperfections and dirt, which prevent it from being used directly to develop composite laminate. To improve the strength, we must go through the chemical treatment process. A lot of chemical treatments occur, such as silane, calcium hydroxide, sodium hydroxide, peroxide, and so on. Based on the literature, NaOH is the best treatment for the other chemical treatment processes. Fig 2. shows the NaOH treatment of the Paddy straw fibers. Sodium hydroxide (NaOH) chemicals were purchased from the go green products at Chennai. 5% NaOH concentration and 24 Hours of soaking time for the paddy straw treatment process [48]. Alkali treatment roughens the surface of the fibers and creates pores. This better surface roughness and porosity facilitate better mechanical intertwining and adhesion between the reinforcement and the matrix.



Fig. 2: NaOH treatment of the Paddy straw fibers

CHARACTERIZATION OF FIBERS

Single fiber test

Single fiber test Preload: 0.1 N; Test speed: 60mm/min; ASTM standard D 3822: Zwick-Roell Z010: 10 KN tensile testing machine. 10 filaments were tested, and average values of force, tenacity, and strain values were measured.

X-Ray Diffraction

A Rigaku Ultima3 X-ray device was used to collect X-ray diffraction data, and powder was used to produce the samples [15]. Using the XRD findings, an evaluation of both untreated and treated fiber performance was conducted by determining the levels of cellulose I and the crystallinity index. The crystalline index was calculated using equation (1). where, CI means Crystalline index, I_{cr} - crystalline intensity region at a 2 θ angle, and I_{am} - amorphous intensity region at a 2 θ angle.

$$CI = \frac{Icr - Iam}{Icr} \times 100 \quad \dots \dots (1)$$

Fourier Transform Infrared Analysis

In the realm of fiber reinforced plastics, infrared spectroscopy stands as a pivotal analytical technique. By detecting molecular vibration, the Fourier Transform Infrared Analysis (FTIR) technique serves as an outstanding method to ascertain functional group scattering and bond types in composites. The examination utilizes a Perkin Elmer RX I apparatus, examining the FTIR spectra of both untreated and treated fibers across the wavenumber range from 4000 to 500 cm⁻¹. The analysis employs a resolution of 4 cm⁻¹ for 32 scans [35,41].

Thermo gravimetric analysis

Evaluating the aptness for applications in elevated temperatures mandates an investigation into the thermal degradation characteristics of composites reinforced with natural fibers. Thermogravimetric analysis spanned from 35°C to 600°C, employing a heating rate of 10°C/min. Sustaining an inert atmosphere, N₂ gas was consistently introduced into the heating systems at a mass flow rate of 20 ml/min.

Composite fabrication

Semi-automatic compression moulding with dimensions of 300mm x 300mm x 3mm was used [33,51]. A weight percentage matrix and reinforcement ratio of 70:30, 60:40, and 50:50 has been used. Randomly oriented fiber was used to fabricate the laminate. The amalgamation of polyester resin, catalyst, and accelerator in a proportion of 1:0.015:0.015, respectively was meticulously agitated for 2-3 minutes to prevent agglomeration [35]. The fibers, arranged in a random orientation, were inserted into the mold container, and the amalgamated resin was poured onto the fibers within the mold container. The temperature of 50°C and a pressure of 30 bar were applied for 45 mins. After 45 minutes, the fabricated laminate was placed at room temperature. This process produced the same results in the other 60:40 and 50:50 ratios. When the fiber percentage increases, the applied pressure also increases. A set of six combinations of composite plates were fabricated and tabulated as shown in Table 1 (where UT means Untreated and ALT means Alkali treated).

S.No	Name of the composite	Composition (wt %)	
	Ivanie of the composite	Resin	Fiber
1	UT-1	70	30
2	UT-2	60	40
3	UT-3	50	50
4	ALT-1	70	30
5	ALT-2	60	40
6	ALT-3	50	50

Table 1: Compositions of composites

Testing of composites

The composite plates, meticulously crafted, underwent precision cutting to adhere to the specified ASTM dimensions, facilitating the creation of specimens for tensile (ASTM D638-08), flexural (ASTM D790-15), impact (ASTM D256-10), shear (ASTM D3846-08), and hardness testing. Employing a universal testing machine (Tinus Olsen H10 KL) with a crosshead speed of 1.5 mm/min, both tensile and three-point bending tests were conducted. The Izod impact test machine, featuring a pendulum design with a 25-joule capacity, was utilized. Each test involved five distinct samples, and meticulous recording of average values ensued. Image of the tensile test before and after specimen shown in Fig 3.



Fig.3: Photographic images of tensile test fractured specimens a) before b) after Surface topography image

Scanning electron microscopy (SEM) stands as the optimal experiment for scrutinizing the morphology of fiber surfaces within fractured composite materials following testing. The SEM equipment employed for this purpose is the Thermoscientific Apreo S, and it is employed to

capture surface images of both untreated and treated fibers, as well as to inspect fractured specimens post-testing.

RESULT AND DISCUSSION

Single fiber test of raw and treated fiber

The Single fiber fragmentation test has several significant practical benefits, including ease of sample preparation and the ability to collect numerous fragments from a single specimen. Single fiber test, raw and treated, is shown in Fig 4. 10 single fibers were taken for testing, and the average readings are noted down in Table 2. Fiber tenacity is also approximately the same in raw and treated fiber. Elongation values achieved in raw and treated fiber are 1.2 and 2.0 % respectively. It is observed that the maximum allowable forces for raw fiber and treated fibers are 106.892 N and 83.748 N, respectively. The application of NaOH led to an augmentation of the fiber surface area. Moreover, this treatment proficiently eliminated specific quantities of lignin and hemicelluloses from the fiber surface [47]. The augmentation in the fiber surface resulted in a broader contact region between the fiber and the matrix. This expansion facilitated an increased number of potential reaction sites, allowing the hydroxyl groups on the cellulose fibers to more efficiently collaborate with the sodium hydroxide-coupling agent [9]. The single fiber tensile strength decreased after surface treatment due to the chemical degradation (degradation can weaken the fiber and may slightly reduce its single fiber strength) and changes in crystallinity (Altering the crystallinity may disrupt the fiber's ability to withstand tensile forces, causing a decrease in single fiber strength).



Fig. 4: Single fiber test – Raw and treated fiber

Table 2: Fiber statistics report

	F _{max} (gf)	Tenacity (g/den)	dL at F _{max} (%)
Raw fiber	10900	1.14	1.2
Treated fiber	8540	1.10	2.0

X-ray Diffraction analysis of raw and treated fibers

X-ray diffraction analysis of raw and treated paddy straw fiber is shown in Fig 5. The chemical alteration induced by alkali treatment involves the breakdown of cellulose chains, thereby augmenting the abundance of hydroxyl (OH) groups on the fiber surface. This augmentation generates a surplus of active sites conducive to chemical bonding with the polymer matrix, ultimately enhancing interfacial adhesion. A solitary prominent peak was detected in the diffractogram, corresponding to specific crystallographic planes. The regions between crystalline peaks and the amorphous curve were compared in calculating the crystallinity index [15]. From the graph, it was found that the Raw paddy straw fibers exhibit a high peak at $2\theta = 22.57^{\circ}$ at the crystallographic plane of (752) and $2\theta = 16.28^{\circ}$ at the (501) crystallographic plane. The Crystalline index was calculated as 33.37% [50]. From the graph, it was found that the Treated paddy straw fibers exhibit a high peak at $2\theta = 22.57^{\circ}$ at the crystalline index was calculated as 33.37% [50]. From the graph, it was found that the Treated paddy straw fibers exhibit a high peak at $2\theta = 22.57^{\circ}$ at the crystallographic plane. The Crystallographic plane of (2748) and $2\theta = 18^{\circ}$ at the (2050) crystallographic plane. The Crystalline index was calculated as 25.40%.



Fig. 5: X-ray diffraction analysis of Raw and treated paddy straw fiber

FTIR analysis of raw and treated fibers

FT-IR analysis of Raw and treated paddy straw fibers is shown in Fig 6. Utilizing FTIR aids in discerning the functional groups within the fiber, accentuating the chemical distinctions among its constituents. The Fourier Transform Infrared spectra, scrutinized within the 4000 to 500 cm⁻¹ range, unveiled a prominent absorption peak at 1028 cm⁻¹. This peak signified the existence of polysaccharides groups, specifically (CO) and (OH), within the cellulose contents. [48,41]. The occurrence at 1240 cm⁻¹ manifests the CO stretching vibration of the acetyl group, aligning with findings akin to those observed by Sabarinathan et al [35]. Verification of wax contents (CC stretching) interspersed within the fiber was established through peaks registering at 2918 and 2857 cm⁻¹, affirming the presence of cellulose and hemicellulose in paddy straw fiber. The peak discerned at 1612 cm⁻¹ is attributed to the stretching of the C=O group within hemicellulose components. The spectral span ranging from 1439 to 1252 cm⁻¹ is linked to the C=H group inherent in lignin constituents [52]. These phenomena indicate that the alkali treatment has extracted lignin, pectin, and hemicellulose from the paddy straw fibers.



Fig. 6: FT-IR analysis of Raw and treated paddy straw fiber Microstructure analysis of the raw and treated Paddy straw

Recorded using a scanning electron microscope, surface images were captured at various magnifications for the morphological study. SEM analysis of raw and treated paddy straw fibers is revealed in Fig 7. Raw paddy straw fibers contain some surface debris and contain a higher amount of bound hemicellulose and cellulose [22]. Treated paddy straw fibers show a very smooth and rough surface. The alkali treatments diminish cellulose and hemicellulose content, fostering an optimal bond among the fiber and the matrix material in the preparation of composites [3].



Fig. 7: Surface topography a) Untreated fiber b) Treated fiber

Thermo Gravimetric Analysis (TGA) of raw and treated fiber

Figure 8 (a) and (b) illustrate the TGA analysis of raw and treated paddy straw fiber, respectively. The customary thermal breakdown of natural fibers at heightened temperatures encompasses multiple elements, such as hemicellulose, cellulose, lignin, wax, and assorted constituents. The thermogram can be segmented into three clearly delineated regions. The initial region (Region I), occurring between 205°C and 278°C, is known as the initial weight loss region or initial shoulder peak. Hemicellulose breakdown is primarily linked to the first phase. The decomposition peak in Region II, which exhibits the greatest weight loss, happens between 278°C and 346°C. The thermal depolymerization of the remaining hemicelluloses, lignin, and cellulose is the cause of this weight loss. And lastly, the third stage of weight loss is represented by Region III, which runs from 346°C to 700°C. This stage is linked to the material's non-living components and the subsequent disintegration of cellulose glucosidic chains.



Fig. 8 (a): TGA analysis of raw paddy straw fiber



Fig. 8(b): TGA analysis of treated paddy straw fiber

MECHANICAL PROPERTIES OF PADDY STRAW FIBER REINFORCED COMPOSITES Tensile strength

Tensile strength values for different wt % of the paddy straw are shown in Fig 9. NaOH treated composite of 50 wt (%) with the tensile strength of 20 MPa is 0.75 and 0.85 times higher than the ALT-1(70%) and ALT-2 (60%), respectively. Comparing the tensile strength, NaOH treated composites show higher value than that of untreated composites [30].

The findings suggest a rise in tensile strength with the elevation of fiber weight percentage, attributed to robust interfacial bonding and excellent adhesive properties in both reinforcement and matrix materials. The main objective of this study is more content of reinforcement and less amount of matrix used. The enhanced individual fiber strength and heightened surface roughness resulting from NaOH treatment played a pivotal role in elevating the tensile performance of all NaOH-treated fiber-reinforced composites, surpassing that of their respective untreated counterparts [35].



Fig. 9: Tensile strength values for different wt % of the paddy straw

Flexural strength

Flexural strength values for different wt % of the paddy straw are shown in Fig 10. Similarly, the flexural strength increases with the increment in fiber weight percentage. NaOH treated composite of 50 wt (%) has flexural strength 31 MPa which is 0.67 times and 0.93 times higher than the flexural strengths of ALT-1 and ALT-2, respectively. As comparing the bend strength, NaOH treated composites show greater value than the untreated wt (%) composites [31]. The results suggest that the flexural strength diminishes because of the uneven dispersion of fibers and inadequate adhesive properties between the reinforcement and matrix [48]. The diminished flexural attributes stem from inadequate resin flow within the composite when a lower fiber content (30 wt%) is present [34]. The introduction of grafted molecules during NaOH treatment resulted in a reduction of voids and interstices on the fiber surface. The removal of impurities, including hemicellulose, wax, oil, pectin, lignin, and environmental pollutants, increased surface roughness and improved the interfacial bonding between the fiber and the matrix [48,50,56].



Fig. 10: Flexural strength values for different wt % of the paddy straw

Impact strength

The abrupt application of a load is a significant factor in the engineering of the material. Impact strength values for different wt % of the paddy straw are shown in Fig. 11. The impact strength increases as the fiber weight percentage is added to the matrix, which also enhances the stiffness and hardness of the composites, demonstrating good adhesive bonding between the reinforcement and matrix [31,32]. NaOH treated composite of 50 wt (%) has impact strength (62 KJ/m²) 0.645 times and 0.548 times higher than the impact strengths of ALT-1 (40 KJ/m²) and ALT-2 (34 KJ/m²). The reduction in impact strength is attributed to the presence of voids, pores, and suboptimal interfacial bonding between the fiber and matrix. The calculation of impact strength depends on the energy required to fracture the sample, contingent upon various factors such as the resilience of the matrix, the shape of the fiber surface, fiber quality, and the crystalline characteristics of both the fiber and matrix [21]. The envisaged transmission of stress between the paddy straw reinforcement and polyester matrix is anticipated to exhibit enhancements in comparison to untreated fibers [47].



Fig. 11: Impact strength values for different wt % of the paddy straw

Hardness strength

Hardness strength values for different wt % of the paddy straw composites is shown in Fig 12. ALT-3 poses 34 HV results are better than the other weight percentages of the samples. The vicker hardness diamond tool measures the penetration of a specified indentor into the specimen. ALT-1 Composite specimen vickers hardness trail value for 13, 19, and 16 HV, respectively. An average hardness value of 16 HV was observed. ALT-2 composite specimen: three trail values were observed in the range of 22, 28, and 25 HV, respectively. The average values of 25 HV and ALT-3 composite specimen trail tests were noticed in the range of 35, 34, and 33 HV, respectively. The average value is 34 HV. Untreated fiber hardness values had limited strength due to the poor adhesive bonding between the reinforcement and matrix. A rougher surface can provide better interlocking and adhesion in composites, improving the strength and hardness of the resulting material. Alkali treatment breaks down the amorphous parts of the fiber, exposing more crystalline regions and aligning the cellulose molecules, thereby enhancing strength and hardness.



Fig. 12: Hardness strength values for different wt % of the paddy straw

Shear strength

The shear strength is a measure of a composite material's resistance to forces that tend to move parallel layers relative to one another. Figure 13 shows the different wt % of the paddy straw. Fig 16 reports that NaOH treated composites 50 wt % shear values are higher than the other compositions of samples. Increase of the fiber content and reduction of the resin content result in increase of the shear values gradually. The maximum strength of the composites under shear strength tests was found to be enhanced by the increase in reinforcement content in the matrix. The untreated shear strength values are 11 MPa, 14 MPa, and 18 MPa, respectively. The supreme shear strength values are achieved in ALT - 3 sample, which are 2.16% and 1.5% higher than the ALT-1 and ALT-2 samples respectively. The composite treated with NaOH exhibits improvements in all mechanical properties in each case, primarily owing to the removal of substances that adversely affect strength, including hemicellulose, lignin, wax, and various impurities. [47]. The whole mechanical behaviours of the paddy straw are shown in Table 3.



Fig. 13: Shear strength values for different wt % of the paddy straw

S.No	Laminate name	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (KJ/m ²)	Shear strength (MPa)	Hardness strength (HV)
1	UT-1	9.5	12	22	11	11
2	UT-2	10.5	15.5	26	14	13
3	UT-3	12	17	32	18	14
4	ALT-1	15	21	40	18	16
5	ALT-2	17	29	34	26	25
6	ALT-3	20	31	62	39	34

Table 3: Overall mechanical properties of the paddy straw

SEM analysis of fractured surfaces

The paddy straw fibers exhibited improvements in mechanical strengths due to the elimination of hemicelluloses and some surface contaminations in the alkali treatment process [17]. The surfaces of the alkali-modified fibers displayed a more rugged and pristine texture in contrast to the untreated fiber surfaces. This finding serves as proof of heightened interfaces and connections between the matrix and the fibers [42]. The treated composite samples

indicated a strong fiber-to-matrix connection with little pull-out or fiber breaking. Fig 14 (a,b) shows micrograph inference on low and high tensile strength fractured specimens using SEM. It observed good fiber surface, fiber breakage, Matrix unfilled area, fiber splitting, and pot holes. From the SEM image of Fig 15 (a,b) reported that fractured surfaces after flexural testing, it is observed that cavity, matrix unfilled area, fiber crack, and pot hole. The micrograph inference on impact fractured specimens are shown in Fig 16 (a,b). In this figure shows that many defects are observed, such as splitting of PALF, Agglomeration of fibers, matrix unfilled areas, voids, and fiber breakage due to impact load. The matrix heavily wets the fibers, and there is fiber breakdown due to high adhesion, which increases impact strength [42,48].



Fig. 14 (a,b): Micrograph inference on low and high tensile strength fractured specimen



Fig. 15 (a,b): Micrograph inference on low and high flexural strength fractured specimens



Fig. 16 (a,b): Micrograph inference on low and high impact strength fractured specimens

Water absorption

Evidently, the water absorption rates for the hybrid composite samples were initially high, then decreased before reaching saturation. Upon saturation, PSUT-3 unveiled the highest water absorption, followed by PSUT-1 and PSUT-2. The highest amount of water absorbed by the PSUT-3 was 49.7%. The adding of fiber content slightly increased the moisture absorption in the presence of composite when compared with PSUT-2 and PSUT-3. Paddy straw fiber indicates higher water absorption content [43]. The water absorption rate for different wt% of the paddy straw is shown in Fig 17. The minimum amount of water absorption rate is PST-1 38.94%. Paddy straw fiber begins to absorb water molecules when it is exposed to water because of its hydrophilic nature. Alkali treatment reduces the hydrophilicity of the fibers by modifying the surface chemistry and reducing the number of hydroxyl groups. This can help reduce water absorption and improve the dimensional stability of the composite. The brittle thermosetting resin micro cracks as a result of this occurrence. The abundance of cellulose in paddy straw fiber aids in enhancing the absorption of water molecules at the micro crack contact. The emergence of swelling stress results in fiber swelling. The consequence was that the matrix failed. Just after the matrix cracks, the water molecules start to form capillaries through the micro crack [7, 26].



Fig. 17: Water absorption rate for different wt % of the paddy straw

CONCLUSION

This paper discussed the characterization of fiber, mechanical, morphological, and water absorption properties. The significant results are listed below:

- Paddy straw fibers were gathered from agricultural land, and an alkali treatment process was conducted to enhance the mechanical performance of the fibers.
- The evaluation of raw and alkali-treated fiber efficacy was conducted through XRD data. FTIR analysis was employed to discern chemical groups linked to cellulose (O-H), hemicellulose (C=O), lignin (C-H), and wax (C=C) constituents. TGA and DTG diagrams were employed to appraise the thermal stability of paddy straw fiber, unveiling the degradation of hemicellulose contents at 234 °C, and cellulose contents at 325 °C. According to SEM findings, the fibers of paddy straw are porous and have a semi-smooth surface, as well as wax content, cellulose content, and hemicellulose content.
- The randomly oriented paddy straw fiber reinforced polyester composites were developed using a semi-automatic compression moulding machine with different weight ratios. For successive wt%, a 50:50 ratio achieved the overall mechanical tests (Tensile, flexural, impact, hardness, shear) of 20 MPa, 31 MPa, 62 KJ/m², 34 HV, and 39 MPa, respectively. From the fractured specimen's micrograph, some defects were observed, such as voids, matrix unfilled areas, pull-out, fiber cracking, and so on.

- The water absorption 30 wt % (ALT-1) fiber content property is recorded at 38.94 %. It is lower than the other wt% of the laminate. Although there is a slight variation in the other ALT 2 and ALT-3 laminates.
- The overall results revealed that the paddy straw fiber is a very prominent material in non-structural applications such as table tops, writing pads, two-wheeler number plates, door panels, partition boards, etc.,

Declaration of competing interests

The authors explicitly declare the absence of any conceivable conflicts of interest concerning the research, authorship, and/or publication of this article.

Funding

The author(s) have revealed that financial backing for the research, authorship, and publication of this article was provided through the Innovation, Technology, Development, and Deployment (1819) initiative of the Department of Science and Technology (DST) under **Reference Number: DST/TDT/WM/2019/78 Consortia (G).**

Acknowledgement

The authors would like to express their gratitude to Dr Krishna Kanth Pulicherla, Scientist, TDT Division, DST, Delhi, India, for their valuable suggestion and support for this research. The authors would like to express sincere gratitude to Dr.Rakesh Kumar Jain, United Nations Industrial development Organization, New Delhi for their mentorship and guidance throughout the research process. Their insights and advice significantly shaped the direction of this work.

REFERENCES

- [1] Ramamoorthy SK, Skrifvars M, Persson A. <u>A review of natural fibers used in bicomposites:</u> <u>Plant, animal and regenerated cellulose fibers</u>. *Polym. Rev.* Jan 2;55(1):107-62 (2015).
- [2] Syduzzaman M, Al Faruque MA, Bilisik K, Naebe M. <u>Plant-based natural fiber reinforced</u> <u>composites: a review on fabrication, properties and applications</u>. *Coatings*. Oct 13;10(10):973 (2020).
- [3] Ahmad F, Choi HS, Park MK. <u>A review: natural fiber composites selection in view of mechanical, light weight, and economic properties.</u> *Macromol. Mater. Eng.* Jan;**300(1)**:10-24 (2015).

- [4] NS, Balaji. <u>Influence of Fabrication Parameters on Thermo-Mechanical Characteristics of</u> <u>Zea Natural Fiber Reinforced Polymer Composites</u>. *Iran. J. Chem. Chem. Eng.* **42(2)**: 437-448 (2022):
- [5] Guzmán- Puyol S, Heredia A, Heredia- Guerrero JA, Benítez JJ. <u>Cutin- Inspired Polymers</u> and Plant Cuticle- like Composites as Sustainable Food Packaging Materials. Sustainable. food pack. technol. Mar 8:161-98 (2021).
- [6] Devireddy, Siva Bhaskara Rao, and Sandhyarani Biswas. "Thermo-physical properties of short banana-jute fiber-reinforced epoxy-based hybrid composites. Proceedings of the Proc. Inst. Mech. Eng., Part L: J. Mater.: Des. Appl., 232 (11): 939-951 (2018)
- [7] Prithivirajan R, Jayabal S, Sundaram SK, Sangeetha V. <u>Hybrid bio composites from agricultural residues: mechanical, water absorption and tribological behaviors</u>. J.Polym. Eng. Sep 1;36(7):663-71 (2016)
- [8] Khan MZ, Srivastava SK, Gupta MK. <u>Tensile and flexural properties of natural fiber</u> reinforced polymer composites: A review. J. Reinf. Plast. Compos. Dec;37(24):1435-55 (2018)
- [9] S. Sathiyamurthy, V. Vinoth, S. Saravanakumar and P. Devi, <u>Investigation On Mechanical</u> <u>Behaviour Of Calcium Carbonate And Groundnut Shell Filler Added Cocos Nucifera Fiber</u> <u>Reinforced Polyester Composites</u> ARPN J Eng and App Sci, **18** (**12**), 1425-1432, (2023).
- [10] Athijayamani A, Thiruchitrambalam M, Natarajan U, Pazhanivel B. Effect of moisture absorption on the mechanical properties of randomly oriented natural fibers/polyester hybrid composite. Mater. Sci. Eng. A. Aug 20;517(1-2):344-53 (2009)
- [11] Ahmadizadegan, Hashem. <u>"Polyester/SiO₂ nanocomposites: gas permeation, mechanical, thermal and morphological study of membranes."</u> *Iran. J. Chem. Chem. Eng.***39(2)**: 33-47 (2020).
- [12] Radzi AM, Zaki SA, Hassan MZ, Ilyas RA, Jamaludin KR, Daud MY, Aziz SA. <u>Bamboo-Fiber-Reinforced thermoset and thermoplastic polymer composites: A review of properties, fabrication, and potential applications.</u> *Polyme.* Mar 29;14(7):1387 (2022)
- [13] Khan MI, Umair M, Hussain R, Karahan M, Nawab Y. <u>Investigation of impact properties</u> of para-aramid composites made with a thermoplastic-thermoset blend. J. Thermoplast. Compos. Mater. Feb;36(2):866. (2023)

- [14] Li, J. "Effect of fiber surface treatment on wear characteristics of carbon fiber reinforced polyamide 6 composites. Iran. J. Chem. Chem. Eng. Research Article 29(1):1-147 (2010):
- [15] Liu Y, Hu H. X-ray diffraction study of bamboo fibers treated with NaOH. Fibers Polym. Dec; 9:735-9 (2008).
- [16] Soundhar A, Kandasamy J. <u>Mechanical, chemical and morphological analysis of crab</u> <u>shell/sisal natural fiber hybrid composites.</u> J. Nat. Fibers. Oct 3;18(10):1518-32 (2021).
- [17] Krishnan T, Jayabal S, Krishna VN. <u>Tensile, flexural, impact, and hardness properties of alkaline-treated Sun hemp fiber reinforced polyester composites.</u> J. Nat. Fibers. 17(3): 326 -336 (2018).
- [18] Saravanakumar, S., S. Sathiyamurthy, P. Pathmanaban, and P. Devi. <u>Integrating machine</u> <u>learning and response surface methodology for analyzing anisotropic mechanical</u> <u>properties of biocomposites</u>. *Compos Interface* 1-28; (2023).
- [19] Fernandes H, Zhang H, Ibarra-Castanedo C, Maldague X. Fiber orientation assessment on randomly-oriented strand composites by means of infrared thermography. Compos. Sci. Technol. Dec 16;121:25-33 (2015).
- [20] Vinoth V, Sathiyamurthy S, Ananthi N, Elaiyarasan U. <u>Chemical treatments and mechanical characterisation of natural fiber reinforced composite materials-a review.</u> *Int. J. Mater. Eng. Innov*.13(3):208-21 (2022).
- [21] Gurukarthik Babu B, Prince Winston D, SenthamaraiKannan P, Saravanakumar SS, Sanjay MR. <u>Study on characterization and physicochemical properties of new natural</u> <u>fiber from Phaseolus vulgaris</u>. J. Nat. Fibers. Oct 3;16(7):1035-42 (2019).
- [22] Moshi AA, Ravindran D, Bharathi SS, Indran S, Saravanakumar SS, Liu Y. Characterization of a new cellulosic natural fiber extracted from the root of Ficus religiosa tree. Int. J. Biol. Macromol. Jan 1;142:212-21 (2020).
- [23] Sathiyamurthy S, Vinoth V, Ananthi N, Devi P. <u>The effect of fiber stacking sequence on</u> mechanical and morphological behavior of paddy straw/pineapple leaf fiber-reinforced ortho-laminated polyester hybrid composites. Proc. Inst. Mech. Eng., Part E: J. Process Mech. Eng. Apr 9:09544089231165943 (2023)

- [24] Saravanakumar, S., S. Sathiyamurthy, V. Vinoth, and P. Devi. <u>Effect of Alumina on Epoxy</u> <u>Composites with Banana Fiber: Mechanical, Water Resistance and Degradation Property</u> <u>Analysis</u>. *Fib and Polym*, 1-13, (2023).
- [25] Balaji NS, Jayabal S. <u>Artificial neural network modeling of mechanical behaviors of zea</u> <u>fiber-polyester composites.</u> Proc. Inst. Mech. Eng., Part E: J. Process Mech. Eng. Feb;230(1):45-55 (2016).
- [26] Sekar S, Suresh Kumar S, Vigneshwaran S, Velmurugan G. Evaluation of mechanical and water absorption behavior of natural fiber-reinforced hybrid biocomposites. J. Nat. Fibers. May 4;19(5):1772-82 (2022).
- [27] Pothan LA, Mai YW, Thomas S, Li RK. <u>Tensile and flexural behavior of sisal fabric/polyester textile composites prepared by resin transfer molding technique</u>. J. Reinf. Plast. Compos. Nov;27(16-17):1847-66 (2008).
- [28] Andre NG, Ariawan D, Mohd Ishak ZA. <u>Mechanical properties and micromechanical analysis of nonwoven kenaf fiber/epoxy composites produced by resin transfer moulding.</u> J. Compos. Mater. Jun;51(13):1875-85 (2017).
- [29] Reddy RS, Mohana Krishnudu D, Rajendra Prasad P, Reddy PV. <u>Alkali treatment</u> influence on characterization of setaria italic (Foxtail Millet) fiber reinforced polymer <u>composites using vacuum bagging.</u> J. Nat. Fibers. May 4;19(5):1851-63 (2022).
- [30] KG A, K K. Mechanical, ballistic impact, and water absorption behavior of luffa/graphene reinforced epoxy composites. Polym. compos. Nov;41(11):4716-26.
- [31] Ashok KG, Kalaichelvan K, Damodaran A. Effect of nano fillers on mechanical properties of luffa fiber epoxy composites. *J.Nat. Fibers*. 2022 Apr 3;**19**(**4**):1472-89 (2020).
- [32] Ashok KG, Kani K. Experimental studies on interlaminar shear strength and dynamic mechanical analysis of luffa fiber epoxy composites with nano PbO addition. J. Ind. Text. Jun;51(3_suppl):3829S-54S (2022).
- [33] Ramesh G, Subramanian K, Sathiyamurthy S, Jayabal S. <u>Study on influence of fiber length</u> and fiber weight proportion in mechanical behavior of Calotropis giganteafiber-epoxy <u>composites</u>. *Polym. Compos.* Nov 1;41(11):4899-906 (2020).

- [34] Ramesh G, Subramanian K, Sathiyamurthy S, Prakash M. <u>Calotropis Gigantea fiber-epoxy</u> composites: Influence of fiber orientation on mechanical properties and thermal <u>behavior</u>. J.Nat. Fibers Oct 3;19(10):3668-80 (2022).
- [35] Muthu Chozha Rajan B, Indran S, Divya D, Narayanasamy P, Khan A, Asiri AM, Nagarajan S. <u>Mechanical and Thermal Properties of Chloris barbata flower fiber/Epoxy</u> <u>Composites: Effect of Alkali treatment and Fiber weight fraction.</u> J.Nat. Fibers. Sep 2;19(9):3453-66 (2022).
- [36] Ismail MR, Yassen AA, Afify MS. <u>Mechanical properties of rice straw fiber-reinforced</u> <u>polymer composites.</u> *Fibers and Polym.* Aug;**12**:648-56 (2011).
- [37] Ming- Zhu P, Chang- Tong M, Xu- Bing Z, Yun- Lei P. Effects of rice straw fiber morphology and content on the mechanical and thermal properties of rice straw fiberhigh density polyethylene composites. J. Appl. Polym. Sci. Sep 5;121(5):2900-7 (2011).
- [38] Dinh Vu N, Thi Tran H, Duy Nguyen T. <u>Characterization of polypropylene green</u> <u>composites reinforced by cellulose fibers extracted from rice straw</u>. Int. J. Polym . Sci. Article ID 1813847; (2018).
- [39] Kamel S. Preparation and properties of composites made from rice straw and poly (vinyl chloride)(PVC). Polym. Adv. Technol. Oct;15(10):612-6 (2004).
- [40] Akinyemi BA, Okonkwo CE, Alhassan EA, Ajiboye M. <u>Durability and strength properties</u> of particle boards from polystyrene–wood wastes. J. Mater. Cycles Waste Manage. Nov; 21:1541-9 (2019).
- [41] Šoštarić T, Petrović M, Stojanović J, Marković M, Avdalović J, Hosseini-Bandegharaei A, Lopičić Z. <u>Structural changes of waste biomass induced by alkaline treatment: the effect</u> on crystallinity and thermal properties. *Biomass Convers. Biorefin*.:1-1 (2020).
- [42] Hu C, Zhou Y, Zhang T, Jiang T, Zeng G. Effect of fiber modified by alkali/polyvinyl alcohol coating treatment on properties of sisal fiber plastic composites. J. Reinf. Plast. Compos Dec;39(23-24):880-9 (2020).
- [43] Ali-Eldin SS, Abd El-Moezz SM, Megahed M, Abdalla WS. Study of hybridization effect of new developed rice straw mat/glass fiber reinforced polyester composite. J. Nat. Fibers. Aug 3;18(8):1194-206 (2021).

- [44] Tutus A, Killi U, Cicekler M. Evaluation of eggshell wastes in office paper production. Biomass Convers. Biorefin. Apr;12(4):1115-24 (2022)
- [45] Saravanakumar, S., S. Sathiyamurthy, N. Ananthi, and P. Devi. <u>Optimization of drilling</u> <u>characteristics of Al₂O₃ and boiled eggshell filler-added hybrid bio composite from</u> <u>agriculture residue</u>. *Biomass Convers. Biorefin*: 1-15 (2023).
- [46] Madhi, Abbas, and Behzad Shirkavand Hadavand. <u>Bio-based surface modification of wool</u> <u>fibers by chitosan-graphene quantum dots nanocomposites.</u> Iran. J. Chem. Chem. Eng. Research Article Vol 41(7): (2022).
- [47] Zhang X, Wang F, Keer LM. <u>Influence of surface modification on the microstructure and thermo-mechanical properties of bamboo fibers</u>. *Materials*. Sep 24;8(10):6597-608, (2015).
- [48] Sabarinathan P, Rajkumar K, Annamalai VE, Vishal K. <u>Static and dynamic behavior of micrometric agro Prunus amygdalus particulate distributed interpolymer layer- kenaf composite</u>. *Polymer Composites* Aug;**41(8)**:3309-21 (2020).
- [49] Saidani K, Racelma G, Cheraitia K, Lounis A. <u>Elaboration and Study of the Properties of</u> <u>a Polyester Composite Made of Oleaster Fibers Treated and Untreated</u>. J. Nat Fib. Nov 28;19(16):14414-21 (2022).
- [50] Vishal K, Rajkumar K, Sabarinathan P, Arun A. Mechanical and thermal characteristics of steam-exploded silane grafted Kigelia Pinnata fruit (KPF) fiber reinforced vinyl ester polymer composites. Polym Compos. Jun 1; 1:1. (2023) https://doi.org/10.1002/pc.27449
- [51] Balaji NS, Chockalingam S, Ashokraj S, Simson D, Jayabal S. <u>Study of mechanical and</u> <u>thermal behaviours of zea-coir hybrid polyester composites</u>. *Mater Today: Proc.* Jan 1; 27:2048-51 (2020).
- [52] Vishal K, Rajkumar K, Nitin MS, Sabarinathan P. <u>Kigelia africana fruit biofibre</u> polysaccharide extraction and biofibre development by silane chemical treatment. Int J Biolog Macromolecules. Jun 1; 209:1248-59 (2022).
- [53] Davim, J. Paulo. <u>Green Composites: Materials, Manufacturing and Engineering</u>. Berlin, Boston: De Gruyter, (2017).
- [54] Kolli, Murahari and Davim, J. Paulo. <u>Waste Residue Composites</u>, Berlin, Boston: De Gruyter, (2023).

- [55] Kumar, Kaushik and Davim, J. Paulo. <u>Plant and Animal Based Composites</u>, Berlin, Boston: De Gruyter, (2021).
- [56] S. Sathiyamurthy, S. Saravanakumar, N. Ananthi and P. Devi, <u>Optimization of fiber length</u> and filler content for improving the mechanical behaviour of musa acuminate fiberreinforced epoxy composite using response surface methodology, J. Ceram. Proces. Res, 24(4): 683-692, (2023).

CC-ACEQ