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Investigation on the tensile and flexural behavior of coconut inflorescence fiber reinforced unsaturated polyester resin composites

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Abstract

Natural fibers as reinforcement with polymer matrix have gained more advantage owing to its low density, high specific properties, biodegradability and abundant availability. In this connection an investigation has been carried out to make use of a newly identified natural fiber named coconut inflorescence fiber to reinforce with polyester resin. The natural fiber after extraction is subjected to mercerization treatment and the effect of mercerization on the surface of CIF was studied by FTIR and XRD analysis. FTIR spectrum analysis revealed the removal of functional groups present in the natural fiber owing to mercerization of CIF. XRD analysis shows improvement in crystalline size and crystalline index of the CIF as a result of mercerization. Samples of CIF/polyester composites were prepared by varying the length of the fiber as 10, 30, 50, 70 and 100 mm to be reinforced with different volume fractions of fiber/polyester resin as 10, 15, 20, 25 and 30% respectively and the specimens were cut as per ASTM standards for testing. Further samples were taken for testing of tensile strength and flexural strength. Results showed fiber length played a predominant role for the increase in tensile and flexural strength of the composites for 25% volume fraction. SEM analysis was done to predict the effect of fracture behaviour of the composite specimen.

1. Introduction

Natural fibers extracted from various renewable sources are finding prominent role as an alternate to synthetic fiber reinforced polymer composites. The extracted natural fibers as reinforcement with polymer matrix offers more advantages such as reduced density of the products, excellent specific strength and high modulus and also biodegradable [1]. The main drawbacks of natural fibers extracted from renewable sources to be used as reinforcement with polymer matrix are poor dimensional stability and very poor interfacial bonding which is due to hydrophilic tendency of the natural fiber. Therefore natural fibers after extraction from renewable sources are subjected to pretreatment with different chemical agents like KOH, NaOH and KMnO₄ etc., [2]. The natural fibers subjected to chemical treatment results in improved mechanical properties by the removal of lignin, pectin, and primary waxy layers present in the natural fiber. The removal of primary layers from the natural fibers improves the hydrophilic tendency of the fiber thereby interfacial bonding between the fiber and polymer matrix gets improvised. High specific modulus and higher elongation at break makes the chemical treated natural fibers to be potential replacement for synthetic manmade fibers. Therefore it is clear that natural fibers after extraction from renewable sources must be subjected to chemical treatments. Several researchers reported on the various mechanical, thermal and other related properties of coconut fiber namely coir, husk and spathe [3–5] as reinforcement material with polymer matrix. In this connection a new fiber is identified from coconut tree named Coconut Inflorescence Fiber (CIF). The inflorescence fiber extracted from coconut tree exhibits better mechanical properties when compared to other natural fibers which is shown in table 1 [6–10].
Table 1. Comparison of different properties of CIF with other natural fibers.

| Fiber Name                  | Density (kg m$^{-3}$) | Diameter ($\mu$m) | Tensile strength (MPa) | Tensile modulus (GPa) | % of Elongation |
|-----------------------------|-----------------------|-------------------|------------------------|-----------------------|-----------------|
| Coconut Inflorescence Fiber | 1500                  | 320–560           | 200–450                | —                     | 4.1–7.5         |
| Hemp                        | 1480                  | —                 | 550–900                | 70                    | 1.6             |
| Cotton                      | 1600                  | —                 | 287–597                | 5.5–12.6              | 3–10            |
| Ramie                       | 1500                  | —                 | 220–938                | 44–128                | 2–3             |
| Jute                        | 1460                  | —                 | 393–800                | 10–30                 | 1.5–1.8         |
| Sisal                       | 1450                  | 50–300            | 227–400                | 9–20                  | 2–14            |
| Red banana                  | —                     | —                 | 482–567                | —                     | 30.6            |
| Poovan banana               | —                     | —                 | 144–206                | —                     | 21.8            |
| Bamboo                      | 910                   | —                 | 503                    | 35.91                 | 1.4             |
| Kenaf                       | 1400                  | 81                | 250                    | 4.3                   | —               |
| Elephant grass              | 817                   | 70–400            | 185                    | 7.4                   | 2.5             |
| Date                        | 990                   | —                 | 309                    | 11.38                 | 2.73            |

Therefore the proposed work is intended to investigate the possibility of using CIF as reinforcement with unsaturated polyester resin. By this work a new composite material can be developed which is inexpensive, eco-friendly and locally available. The extracted CIF has some disadvantages such as high moisture content, and also the complex nature of fiber results in dimensional instability of the composites when reinforced with polymer matrix. Therefore the extracted CIF before reinforcement with polyester resin is subjected to mercerization. The effect of mercerization on the surface of CIF is evaluated by Fourier Transform Infrared Spectroscopy (FTIR) and x-ray diffraction technique (XRD).

2. Experiments

2.1. Extraction of CIF

Natural fiber used in this work was extracted from inflorescence of coconut tree which is available abundantly in the southern part of India. The process of extracting fiber involves simple biodegradable treatments. The fibers are manually extracted from the coconut inflorescence after subjecting to retting process by which primary and secondary walls are softened owing to biodegradation. Then the inflorescence is crushed with mallet after retting to extract fibers. Finally the extracted fibers were cleaned and dried under sunlight to remove moisture content in the fiber. The process of extracting fibers from coconut inflorescence is shown in figure 1.

2.2. Polyester resin

Isophthalic unsaturated polyester resin which is available commercially is used for this investigation. Methyl Ethyl Ketone (Accelerator) and Cobalt Naphthalene (Catalyst) were used to cure the resin. Owing to its lower water absorbing capability, better flexibility, chemical resistance and excellent bonding tendency, isophthalic polyester resin is selected for this investigation. The properties of thermoset isophthalic polyester resin as specified by the supplier used in this investigation are shown in the table 2.

2.3. Mercerization of CIF

The fiber matrix interface is the critical zone in which two phases are combined chemically/mechanically or by both. The mechanical properties of the developed composites are hindered primarily by interfacial bonding between the fiber/matrix. Hydrophilic tendency, dimensional instability and complex structure inhibit the effective bonding between fiber/matrix. Inaddition fiber inner substance like lignin, pectin, wax which covers the fiber restricts effective bonding between the fiber/matrix. To improvise the interfacial bonding, the extracted CIF were subjected to alkali treatment with 95/05 v/wt% of water/NaOH mixture solution for a fiber measuring of 25 wt% at room temperature for one hour. As a result the hemicellulose, lignin, and pectin, wax and surface impurities present in the fibers will get eroded. Finally the fibers are thoroughly cleaned with distilled water followed by drying under direct sunlight to remove moisture content present in the CIF.

2.4. FTIR analysis

To examine the functional groups present in the untreated and mercerized CIF, Fourier Transform Infrared Spectroscopy analysis (FTIR) was adopted. The untreated and mercerized CIF were converted into powder form and sample preparation for testing is done by KBr pellet method. FTIR measurement was done using a Perkin-
Elmer spectrometer with a resolution of 2 cm$^{-1}$ at a scan rate of 42 scans per minute in the 500–4000 cm$^{-1}$ wave region.

2.5. XRD analysis

X-Ray diffraction studies were performed at 2θ scale ranging from 10° to 60° for both untreated and mercerized CIF in powder form to measure the crystallinity size and crystallinity index using a x-ray diffractometer at an accelerating velocity of 45 Kv and a current of 30 mA with Cu,K$\alpha$ radiation. The sample preparation for XRD test is done based on KBr pellet method.

2.6. Fabrication of composite specimens

Fiber length plays an important role in determining the mechanical properties of the composites during the manufacturing of composites. Therefore any practical experimental work needs to be done to study the impact of fiber length with reference to the applied load on the moulded composites. Therefore the mercerized CIF were cut to different length of 10 mm, 30 mm, 50 mm, 70 mm and 100 mm to study the optimum fiber length for reinforcement with polyester resin. Hand layup technique followed by compression moulding is adopted to fabricate composite samples. The volume fractions followed in this experimental study are 10, 15, 20, 25, 30% of fiber/matrix respectively. The die is sprayed with mould releasing spray for easy removal of composite samples.
after curing. Steel rollers are used to maintain uniform distribution of fibers with the matrix. At the time of curing the moulds are applied a pressure of 10 MPa for 10 min. After curing composite samples are taken out of the mould. Figure 2(a) shows the compression moulding set up used and figure 2(b), shows the fabricated composite sample.

2.7. Tensile testing of composite specimens
Tensile test were conducted for the CIF polyester composites using universal testing machine to obtain required properties. For tensile test dog bone shaped specimen of size 165 mm length, 19 mm width and 3.2 mm thickness as per ASTM D638 standard [11] was followed. The tensile behavior of CIF polyester composite were evaluated with the test parameters of gauge length 100 mm, 2 mm min$^{-1}$ cross head speed and with a 10 000 N load cell. From the experiments conducted tensile strength, tensile modulus, elongation at break was obtained. Figure 3(a) shows tensile test specimen and figure 3(b) shows the tensile test set up used for the experiment.

2.8. Flexural testing of composite specimens
Flexural test were conducted for the CIF polyester composites using 3 point bending flexural test setup. For flexural test specimen of size 76 mm length, 25 mm width and 3.2 mm thickness as per ASTM D790 standard [12] was prepared. The flexural behavior of CIF/polyester composite were evaluated with test parameters of gauge length 40 mm, load cell of 10 000 N and 2 mm min$^{-1}$ cross head speed. Five specimens were tested for each test. From the experiments conducted flexural strength and flexural modulus, elongation at break was obtained. Figure 4(a) shows the sample flexural test specimen and figure 4(b) shows broken specimen between the gauge length during the flexural test experiment.
3. Results and discussions

3.1. Effect of mercerization on tensile strength of CIF

The tensile properties of CIF were tested by Instron_5500R with 100 mm gauge length for a cross head speed of 100 mm min$^{-1}$, 65% humidity, followed by 25°C operating temperature. Thirty samples were tested for both untreated and 5% NaOH treated CIF. The tensile strength of 5% NaOH treated CIF observed to be 331.2 MPa and for untreated CIF it is 256 MPa which is calculated by the ratio of average load to the average area. Mercerization results in rearrangement of fibrils along the tensile deformation direction which further promotes even distribution of load in the fibers thereby reducing the stress concentration. Hence tensile strength of mercerized CIF increases. And also pretreatment of fibers resulted in reduction of hydroxyl group and increase in moisture resistance property of fiber and also stress transfer capacity among the fiber cell also getting improved.

3.2. Effect of mercerization on CIF diameter

The diameter of the fibers were evaluated with the help of image analysis technique by scanning electron microscope at different length along the width of the CIF [13]. Thirty samples were observed for mean, standard deviation and standard error for both untreated and treated fibers [14, 15]. The mean diameter of 5% NaOH treated CIF was 0.42 mm while that of untreated CIF the diameter was found to be 0.51 mm. Axial splitting of fibrils along the length of the CIF was observed which leads to decrease in fiber diameter [16]. Thus enhanced mechanical interlocking of fibers which further leads to a better fiber/matrix adhesion. When the percentage of alkali treatment increased above 5% fiber surfaces are getting damaged which further leads to reduced tensile strength of the CIF.

3.3. FTIR spectrum analysis of CIF

FTIR spectrum analysis is used to examine the effect of mercerization on the surface of CIF. From figure 5 it is observed that higher peak of 3292.33 cm$^{-1}$ indicates OH stretching vibration of hydroxyl band of cellulose I and II [17]. The peak at 2852.70 cm$^{-1}$ in untreated CIF corresponds to C-H symmetrical stretching whereas the peak disappeared in mercerized CIF indicating the removal of wax and other surface related impurities which contribute to better adhesion between the fiber/matrix [18]. Similarly peak at 1737.21 cm$^{-1}$ in untreated CIF corresponds to stretching vibration of carboxyl group, carboxylic acid (RCOOH) in hemicellulose and lignin [19]. The disappearance of 1737.21 cm$^{-1}$ peak in mercerized CIF indicates the removal of hemicellulose and lignin from the mercerized CIF. The peak at 1549 cm$^{-1}$ corresponds to C–C=C aromatic symmetric stretching which corresponds to lignin [20] and peak at 1238 cm$^{-1}$ represents C–O stretching in acetyl group that is acetyl group present in the hemicellulose [21] and both the peaks were disappeared in mercerized CIF which draws to the conclusion on the removal of lignin and hemicellulose in mercerized CIF. Therefore from the FTIR spectrum of untreated and mercerized CIF it is concluded that mercerization of CIF resulted in removal of hemicellulose, lignin, wax and other surface related impurities present in the CIF.

3.4. X-ray diffraction analysis of CIF

The diffraction peak values of crystalline region and amorphous region are presented in figure 6. The peaks of untreated CIF corresponds to crystalline region $I_c$ 29.31 and amorphous region $I_A$ 21.58 whereas for mercerized CIF peaks are represented by crystalline region $I_c$ 27.14 and amorphous region $I_A$ 19.36. The crystallinity index was estimated as 57.59 and 58.36 for untreated and mercerized CIF respectively. It is clear that fiber swelling,
Disruption of crystalline areas and new crystal lattice formations were observed by the mercerization of CIF [22] as a result crystallinity gets improved and thereby making it to use as reinforcement with polyester resin. The cementing materials like amorphous region and cellulose structure is interacting with alkali and getting eroded. Thus interfibrillar regions gets less dense, rigid and rearrangement of fibrils happens by themselves and also the crystallinity index of the mercerized CIF increases slightly as a result of mercerization [23].

3.5. Tensile strength of composite specimens
Figure 7 shows the tensile strength variation for the increased fiber volume fraction of different length of fiber reinforcements. Increase in tensile strength was observed when the fiber length is inflated from 10 mm to 70 mm for 10% of $V_f$. Thus addition of filler material to matrix results in improved stress transfer between the fiber/ matrix [24]. The maximum and minimum tensile strength was observed between 25% and 10% $V_f$, and maximum tensile strength of 72.14 MPa was observed for 70 mm fiber length with 25% of volume fraction. Minimum tensile strength was observed to be 17.19 MPa for 10 mm fiber with 10% of volume fraction. And 15% improvement in tensile strength was observed for 25% $V_f$ of 70 mm fiber length between the minimum and maximum. The critical factor contributing to maximum tensile strength for 70 mm length of fiber is higher fiber.

![FTIR analysis of CIF](image1)

**Figure 5.** FTIR analysis of CIF.

![XRD analysis of CIF](image2)

**Figure 6.** XRD analysis of CIF.
ends this is due to process of retting during the extraction of CIF [25]. As a result of retting external fibrillation occurs which in turn causing the primary walls of the fiber to partially or totally remove causing fibrils forming on the surface of the fiber and therefore fiber surface area getting increased and also fibers become conformable due to internal fibrillation. From the result it is clear that stress transfer capacity between fiber/matrix gets affected due to inadequate matrix in composites. The maximum tensile strength of composite is observed for 70 mm fiber length with 25% of $V_f$. The reason for sudden drop in tensile strength when the volume fraction is 30% is due to inadequate matrix in the composite as the fiber length is high in the composites [26]. Hence stress transfer among the fiber/matrix decreases as a result tensile strength decreases.

Figure 8 shows the tensile stress strain curve for different fiber volume fraction at 70 mm fiber length. The stress strain curve of the unsaturated polyester resin depicts brittle nature while the addition of CIF to the polyester resin converts the brittle effect to ductile nature. The elongation at break of the polyester resin is lower than the 10% $V_f$ composite samples. The composites with 25% $V_f$ contributes to the maximum stress value with an increase in strain. The stress strain curve of 25% $V_f$ contribute to maximum tensile strength of the composites. The young’s modulus of the composite sample is calculated by the elastic nature of stress strain curve. The reason for decrease in stress value when the fiber length is higher is due to intricate nature of the CIF.

Figure 9 shows the tensile modulus over varying fiber volume fraction for different fiber length of CIF/polyester composites. The composite with 10% $V_f$ contribute to tensile modulus of 245 MPa for 100 mm fiber length and minimum tensile modulus of 135 MPa for fiber length of 10 mm respectively. Then the tensile
modulus is increased to 281 MPa for the composites with 15% and when the $V_f$ is 20% tensile modulus is 430 MPa for a fiber length of 70 mm. Then the composite with 25% $V_f$ contribute to the maximum tensile modulus of 475 MPa in the entire investigation for a fiber length of 70 mm. Gradual increase in tensile modulus upto 25% $V_f$ was observed and then the maximum tensile modulus is reduced to 342 MPa for composites with 30% $V_f$. The tensile modulus gets decreased when there is insufficient dispersion of matrix in the composites. This proves that length of fiber and volume fraction has prominent effect in setting the tensile modulus of any natural fiber polymer composites.

3.6. Flexural strength of composite specimens

Figure 10 shows flexural strength variation over different fiber volume fractions for different fiber length. From the figure it is clear that flexural strength increases when the length of fiber is increased with the increase in volume fraction of the composites. The maximum flexural strength is observed to be 122.7 MPa for 100 mm length of fiber with 25% $V_f$, while minimum flexural strength of 30 MPa was observed for 10% $V_f$. When the volume fraction of the fiber is increased above 25% $V_f$ there is insufficient dispersion of matrix with the fibers surrounding as discussed in the previous case which results for fractional composites. As a result the flexural strength decreases for composites with 30% $V_f$. Gradual increase in flexural strength was observed for increase in fiber length upto 25% $V_f$. Then decrease in flexural strength was observed for 30% $V_f$ which leads to the
conclusion that insufficient filling of polyester matrix into the CIF and it is the main reason for fractional composites. Therefore increase in the fiber length of the composite contributes to the maximum flexural strength of the composites [27]. From the figure it is inferred that maximum flexural strength was carried by 100 mm fiber length with 25% V_f. From the experimental data it is clear that long CIF contribute to maximum flexural load rather than the shorter CIF [25].

Figure 11 shows the flexural stress strain curve for different fiber volume fraction at 100 mm fiber length. As discussed earlier the brittleness of the unsaturated polyester resin is converted into ductile nature by the addition of CIF to the matrix which is visible from the flexural stress strain curve. From figure 11 it is also clear that the increase in strain contribute to increase in stress value for a fiber length of 100 mm. Maximum flexural strength is observed for 25% V_f composites which is due to higher fiber ends as discusses earlier. Thus long CIF contribute to the maximum flexural load of the composites rather than the short CIF.

Figure 12 shows the flexural modulus of CIF over varying volume fraction of CIF/polyester composites. As like previous case flexural modulus increases with increase in fiber volume fraction. 25% V_f of the CIF/polyester composite contribute to the maximum flexural modulus of 21.3 GPa when the length of the fiber is 100 mm. Then the maximum flexural modulus is dropped to 12 GPa for 30% V_f. The main reason for sudden drop in flexural modulus is the partial filling of matrix in the composites. This concludes that composites 25% V_f for the fiber length of 100 mm contribute to the maximum flexural modulus. From the results it is inferred that long
CIF contributes to increased flexural strength, flexural modulus of CIF/polyester composites for the composites with 25% $V_f$. One of the contributing parameter for increase in strength is the fiber reinforcement with the polyester matrix is higher fiber ends. It also draws to the conclusion that strength of CIF/polyester composites depends on important factors like fiber diameter, fiber density, fiber length, fiber volume fraction and orientation of fibers and also the CIF have aligned well with higher degree of orientation with the polyester matrix owing to higher fibril ends. Thus the load transfer happens from one end of the fiber to the other end of fiber. Therefore it is concluded that determination of mechanical properties of the CIF/polyester composite samples is possible only by variation of volume fraction with variation in fiber length.

3.7. SEM analysis
SEM analysis was done to ascertain the surface morphology of fractured CIF/polyester composites. From figure 13(a) it is clear that minimum voids are present in the composites. This is due to the compressive force which is applied during the manufacturing of composites. It is also evident that there is good interfacial bonding between the CIF and polyester matrix which can be inferred from the maximum flexural strength of 122 MPa for 100 mm fiber length of 25% $V_f$. The curing of polyester matrix resulted in cater like structure which is due to the catalyst and accelerator reaction taking place. Figure 13(b) shows the fracture surface of 30% $V_f$ CIF/Polyester composites. Fiber pull-outs were observed in the composite which was due to very poor interfacial bonding between the CIF and polyester. And also main reason for decrease in flexural strength when the volume fraction increased to 30% is higher fiber ends which further result in poor interfacial bonding between the fiber and the matrix.

4. Conclusion
The present work clearly depicts that lignocellulose CIF can be used for reinforcement with polyester matrix. And as per the experimental study the following conclusions are derived. Extraction of coconut inflorescence fibers (CIF) with low cost and composites can be manufactured via simple techniques. The properties of extracted CIF are superior when compared to other natural fibers available. And also CIF can be used as viable reinforcement in unsaturated polyester matrix owing to its promising mechanical properties. Mercerization treatment of CIF resulted in reduction of hydrophilic tendency of the fiber, improvement in surface morphology and making it as a suitable alternate for synthetic fibers. FTIR spectrum analysis reveals the removal of cellulose, lignin, wax and other surface related impurities present in the CIF due to mercerization. XRD analysis reveals increase in crystalline size and crystalline index of CIF owing to mercerization. Significant increase in tensile strength of CIF and also the mechanical properties of CIF/polyester composite increases over increasing volume fraction and fiber length. Tensile and flexural strength increases when the volume fraction of the fiber increases. The maximum tensile strength of 72.14 MPa was observed for 70 mm length of CIF with 25% volume fraction and the maximum flexural strength of 122 MPa was observed for 100 mm length of fiber with 25% volume fraction. From the results it is clear that 25% volume fraction contribute to maximum mechanical properties of CIF/polyester composites.
the CIG/polyester composites, fiber length plays a predominant role during the manufacturing of composites. SEM analysis reveals about the fiber pullout in the composite specimen when the volume fraction is high. Overall it is inferred that 25% volume fraction contribute to maximum mechanical properties of the composites and also length of the fiber plays a major role in the manufacturing of the composites.

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