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CHARACTERIZATION OF CALOTROPIS GIGANTEA FIBER AND ITS COMPOSITES

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ABSTRACT

The present investigation is focused on the characterization of fibers, Alkaline treatment of fibers, fabrication of Calotropis Gigantea-Epoxy composites and evaluation of tensile, flexural, impact, hardness, water absorption and wear behaviours. The presence of various organic materials in Calotropis Gigantea fiber was identified by using X-ray diffraction technique. The chemical structure of various groups and bonds in untreated and treated fibers were identified using Fourier Transforms Infrared spectroscopy. The thermal behaviors such as weight loss due to temperature and melting point were identified for the fiber and epoxy resin using Thermogravimetry Analyzer/Differential Thermal Analyzer. The tensile properties of raw and treated king crown fibers were evaluated using Instron Universal Testing Machine as per ASTM D638. The composite fabrications were planned as per design of experiments method using full factorial design. The fabrication parameters such as fiber length in three levels and fiber volume in six levels were selected and 18 composites were fabricated.

Keywords: *XRD, SEM, FTIR, TGA, DTA and Calotropis Gigantea.*

1. Introduction

Natural fiber depends mainly on the nature of the plant, locality in which it is grown, age of the plant, extraction and processing methods used. A large number of natural fibers are available in the world, but very few have been exploited and recognized as potential reinforcement materials for polymer composite applications. The interfacial adhesion between fiber and matrix is enhanced with the help of various chemical treatments of fibers. A strong alkaline treatment removes lignin, hemi-cellulose and other alkali soluble compounds from the surface of the fibers to increase the numbers of reactive hydroxyl groups on the fiber surface available for chemical bonding. Moreover, the alkali treatment made the fiber surface clean by the removal of waxes, hemi cellulose, pectin and part of lignin. The removal of these substances enhanced the surface roughness. Therefore, the mechanical interlocking at the interface could be improved.

To begin with, the effect of silane treatment on the physical and mechanical properties of sisal fiber reinforced epoxy composites were carried out Bisanda et al [1]. In this continuation, adding of silane treated wood flour to polypropylene produced a sustained increase in the tensile strength and modulus of the composites Ichazo et al [2]. The significance of surface treatment on the coir fiber reinforced polyester composites were studied Rout et.al [3]. Bleached fiber composite (at 65^{0}C) showed better flexural strength and NaOH treated fiber/polyester composite exhibited better tensile strength were carried out Mohanty et.al [4].

The chemical reaction process on the surface of the fiber to improve the mechanical properties and the structure of fiber constituents studied varga et.al [5].All modifications of the fiber surfaces have increased the mechanical properties of composites Herrera-Franco et al [6-8]. Removal of lignin, hemicelluloses, silica and pith from the fiber to have better impregnation between fiber and matrix and improving fiber surface roughness to have better interactions on the main objectives of fiber alkali treatment were carried out Geethammaet.al [9,10].

The correct balance level of time and concentration of the treatment ensure the optimum mechanical properties of composites mizra et.al [11]. However, there is nothing conclusive in this research area about the concentration and time of treatment with respect to the optimum mechanical properties of composites srinivas et.al [12]. The study of physic-chemical characterization of different treatment

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sequences of kenaf bast fiber of composites vaizanen et.al [13]. The research on the treatment of natural fibers was initiated before preparing polymer composites ganeshan et.al [14]. The treated composite with 25 % fiber content exhibited higher mechanical properties of composites.

2. Experimental Methods

2.1. Materials

Calotropis Gigantea, called as King's crown fiber, is a large shrub and has clusters of waxy flowers that are either white or lavender in colour. The plant has oval, light green leaves and milky stem. The present investigation is initially focused on the extraction and processing of CG fiber from the stem. The CG fibers were extracted from King's Crown stem. Around one year old plants are found to be more suitable for fiber extraction. The leaves attached to the stem are removed and then the stems are cut to the required length. After that they are allowed to dry in room temperature for 2-4 days. The fibers are isolated from the dried stems manually by hand de-cortication method. In hand de-cortication method, the outer bark is initially removed followed by extraction of fiber through scraping with a sharp knife and dried in free air. Finally the fibers are dried at room temperature for 2 days and cut into 10, 20 and 30 mm lengths for the preparation of composite plates. The fiber is white in colour and the extraction of CG fibers is shown in Figure 1. respectively.

Figure 1.Extraction processes of CG fibers,

a) CG Plant, b) Stem of the plant, c) retting of fibers d) extracted fibers.

2.2. Surface modification

The CG crown fiber is rinsed with water and dried at room temperature for two days. The surface modification is carried out by alkali treatment of the fiber with different NaOH concentrations, 5%, 10% and 15% respectively, for 30 minutes. This treatment removes certain amount of lignin, wax and oil covering the external surface of the fiber cell wall. The fiber could be used as a better reinforcement in polymeric composites based on the evaluation of tensile strength of raw and treated fibers using single filament testing.

3. Testing Standard and Equipment

X-ray diffraction study was used to identify the organic materials present in CG fiber. XRD analysis is carried out with a Pan Analytical Expert Pro at Central Electro Chemical Research Institute (CECRI), Karaikudi, India. The constituents of organic materials present in the CG fiber are analyzed using PCPDWIN software by referring diffraction angle of corresponding peak intensity and d-spacing of materials.

Fourier transform infrared spectroscopy (FTIR) spectra are recorded using a Perkin Elmer make red infra spectrometer available at National Institute of Technology, Thiruchirappalli, India. The particles of FTIR spectra are analyzed for chemical structure of various groups and bonds. The functional group and chemical structure of CG fiber are determined using Fourier Transform Infrared Spectroscopy. The thermal behaviour of CG fiber and its composites were determined using STA 600 Perkin–Elmer Simultaneous Thermal Analyzer (A.C. College of Engineering and Technology, Karaikudi, India) by applying different cycles of heating and cooling the monofilament of each fibers is tested using the Instron (Model: 5500 R) Testing machine at the rate of loading of 5 mm/min until the fiber fractures at a temperature of 21 ± 1^0C and at humidity of 55 ± 2 %. The tensile strength of the fiber decreased the optimum with NaOH concentration (5% NaOH).

The result obtained for 10 fiber consignment in single, three and five sample at three levels of chemical treatment, namely raw, 5% NaOH, and 10% NaOH treated are presented in Table 1. The mechanical properties of alkalized fiber depended on the concentration of NaOH. The micro structural failures that occurred in the fractured composite specimens under tensile, flexural and impact conditions are analyzed through a JEOL Scanning Electron Microscope model JSM-6390.

Table 1.Tensile properties of raw and treated CG Fibers

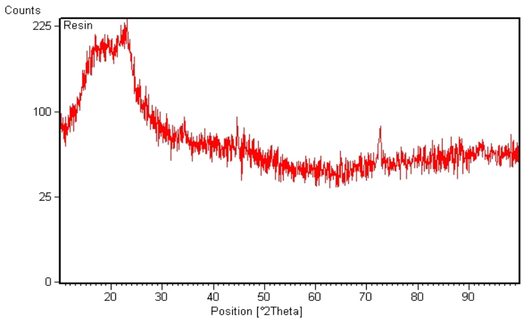
Fiber Type	Tensile Force (N)	CV % of strength	% of elongation	CV % of elongation
Raw fiber	7.95	16.09	5.03	33.32
Treated fiber (5 % NaOH)	8.93	24.08	6.12	30.81
Treated fibe (10 % NaOH)	8.33	23.12	5.39	41.01
Treated fiber(15 % NaOH)	8.15	22.06	4.87	42.31

4. Results and Discussion

4.1. XRD analysis of materials

The amorphous nature of cured epoxy resin is observed by variation of intensities with the scattering angle and the formation of very broad peaks as shown in Figure 2.

X' Pert High Score software is used for crystallographic analysis in order to identify the organic materials present in the material. The presence of carbon, hydrogen and chlorine are identified by XRD peaks in epoxy resin as the chemical formula is $C_{21}H_{25}ClO_5$. The semi crystalline structure is observed by studying XRD pattern of CG fiber and these fibers have orthorhombic symmetry. Crystals in this system are referred to three mutually perpendicular axes that are unequal in length. The presence of sodium and silicon are identified using XRD peaks. The amorphous structure is observed using XRD pattern in CG fiber-epoxy composites. The rhombohedral lattice structure is observed by interpreting XRD data. The properties of fiber are better when compared with composites due to the semi crystalline nature of fibers and amorphous nature of CG fiber-epoxy composites.



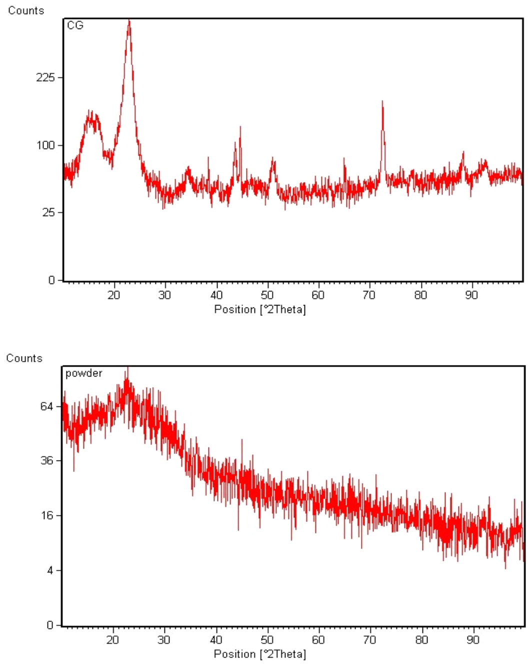
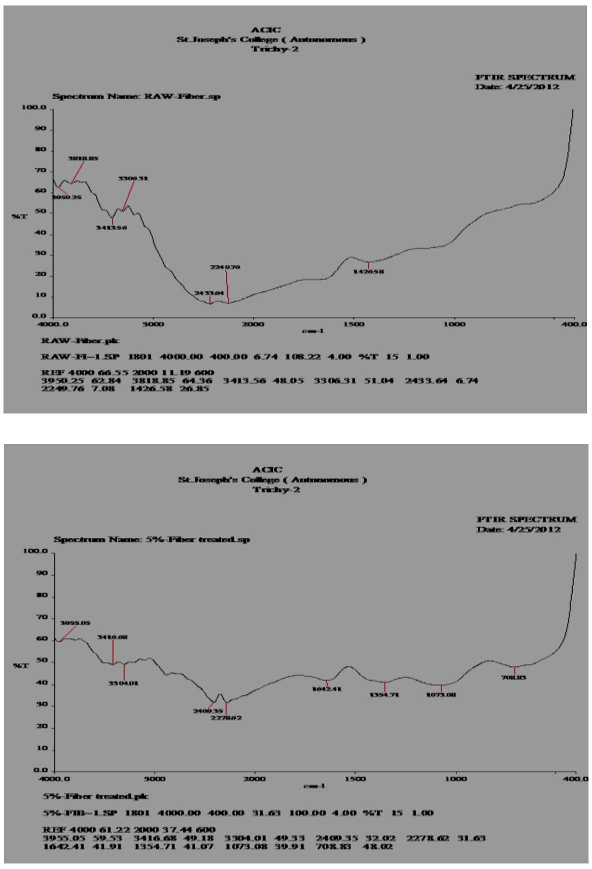


Figure 2. XRD pattern for fiber, resin and composites

4.2. FTIR analysis of materials

The FTIR of raw fiber and NaOH treated fiber are shown in Figure 3. The peak at 2433 cm^{-1} is due to C-H stretching in polysaccharide chains and the peak at 2249 cm^{-1} is due to O-H stretching of absorbed moisture. The peak 1426 cm^{-1} is due to –CH, $-CH_2$ and CH_3 bonding groups. In Alkaline treatments, 5% NaOH shows that there is a small difference in wave number between raw fiber and treated fiber. There also exist new functional groups like halogen at 708 cm^{-1}, C-H vibration of aryl group at 1073 cm^{-1}, C-H and C-O bond in the polysaccharides at 1354 cm^{-1}. It leads to the presence of more active sites in treated fiber and enhances the binding capacity and strength of the composites. The presence of hydroxyl group, OH stretching, OH bending, carbon and oxygen bonding (C=O) and carbon–carbon bonding (C-C) in the 5 % NaOH treated fiber confirms the adhesion characteristics of CG fiber with epoxy resin.



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RAW-Fiber.pk
RAW-FI--1.SP  1801  4000.00  400.00  6.74  108.22  4.00  %T  15  1.00
REF 4000 66.55 2000 11.19 600
3950.25  62.84   3818.85  64.36   3413.56  48.05   3306.31  51.04   2433.64  6.74
2249.76  7.08    1426.58  26.85
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5%-Fiber treated.pk
5%-FIB--1.SP  1801  4000.00  400.00  31.63  100.00  4.00  %T  15  1.00
REF 4000 61.22 2000 37.44 600
3955.05  59.53   3416.68  49.18   3304.01  49.33   2409.35  32.02   2278.62  31.63
1642.41  41.91   1354.71  41.07   1073.08  39.91   708.83   48.02
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Figure 3. FTIR studies for untreated and treated CG fiber.

4.3. Thermal studies of materials

The TGA/DSC curves for the raw and treated fibers are shown in Figure 4. In raw fiber, the initial weight loss of the first stage is observed at temperatures between 50oC and 300oC due to the vaporization of water from the fiber. The degradation of the fiber occurs at high temperature, particularly after 300oC. In the second stage, the temperature between 300°C to 350°C corresponds to the thermal depolymerization of hemicelluloses and the cleavage of glycosidic linkages of cellulose. These results are also confirmed by TGA curve as indicated in blue wherein, the maximum decomposition rates for the weight losses are observed. In 5% NaOH treated fiber, the initial weight loss of the first stage is observed at a temperature between 40°C and 213°C due to the vaporization of water from the fiber and degradation of the fiber occurs at high temperature, particularly after 213°C. In the second stage, the temperature between 213°C to 366°C corresponds to the thermal depolymerization of hemicelluloses and the cleavage of glycosidic linkages of cellulose. Again, thermal degradation takes place from the temperature 347°C to 580°C.The peak temperature of 534.09°C is obtained in treated fiber, whereas 308.02°C is obtained for raw fiber.

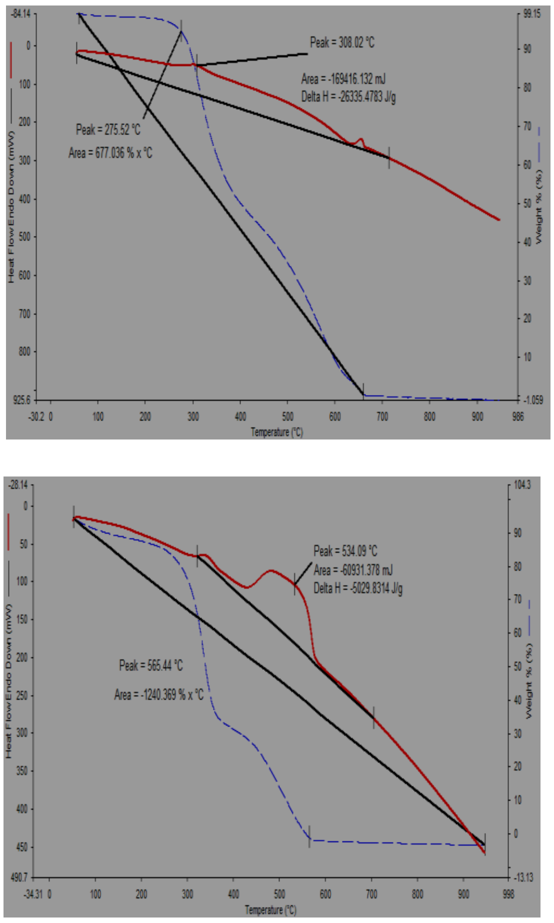

Figure 4. TGA/DTA plots for raw and treated fiber

4.4. Tensile, flexural, impact, hardness, water absorption and specific wear rate of composites

The statistical summary of observed properties is given in Table 2.The comparison of tensile strength of various natural fiber/particulate composites is listed in Table 3. The increase of tensile strength of 76%, 72%, 65% and 52 % is obtained in CG fiber-epoxy composites compared with cured epoxy resin, rice husk-epoxy, coir-epoxy and sisal-epoxy composites respectively. The rice husk-epoxy composites exhibited better value of tensile strength among the different agricultural residues and industrial wastes reinforced with epoxy composites. For the comparison of tensile strength, the two natural fiber-composites such as coir-epoxy and sisal-epoxy are taken. The CG fiber-epoxy composites exhibited better value of tensile strength compared with other natural fiber reinforced epoxy composites.

Table 2. Statistical summary of evaluated properties of CG fiber-epoxy composites

Sl. No	Properties	Maximum	Minimum	Average	Standard deviation	Model
1	Tensile strength (MPa)	30.47	64.61	51.26778	9.687427	Quadratic
2	Flexural strength (MPa)	78.07	145	113.5989	18.01094	Quadratic
3	Impact energy (kJ/m^2)	7.8	16.01	12.455	2.153692	Quadratic
4	Hardness (HV)	14.2	29.5	22.12778	4.891386	Linear
5	Water Absorption (%)	1.8	17.2	10.36667	4.943921	Linear
6	Specific wear rate (mm^3/N	0.01	0.1	0.042278	0.028384	Quadratic

A comparison of flexural strength of various natural fiber/particulate composites is listed in Table 4. The increase of flexural strength of 86%, 79 %, 77% and 69% is obtained in CG fiber composites as compared with cured epoxy resin, rice husk-epoxy, coir-epoxy and sisal-epoxy composites respectively. A better value of impact energy of 16.01 kJ/m^2 is obtained for the fiber length of 30 mm and fiber volume content of 25%. A comparison of impact energy of various natural fiber/particulate composites is listed in Table 5. The impact energy of CG fiber-epoxy composites is lower compared with that of cured epoxy resin, rice husk-epoxy, coir-epoxy and sisal-epoxy composites. The comparison of hardness, water absorption and specific wear rate of various natural fiber/particulate composites is listed in Table 6. A better value of harness and lower values of water absorption and specific wear rate are obtained in CG fiber–epoxy composites. The fiber length of 30 mm and fiber volume content of 30% are taken for the fabrication of composites to evaluate water absorption behavior whereas fiber length of 10 mm and fiber volume content of 5 % were taken for the fabrication of composites to evaluate water absorption and specific wear rate properties.

Table 3. Comparison of tensile strength of natural fiber/particulate composites

Sl. No	Composite	Fiber length (mm)	Particulate content (%)	Tensile strength (MPa)
1	Cured epoxy resin	-	-	15.30
2	Rice husk-epoxy	-	30	17.80
3	Coir-epoxy	30	25	22.50
4	Sisal-epoxy	40	30	31.00
5	CG fiber-epoxy	10	25	64.61

Table 4. Comparison of flexural strength of natural fiber/particulate composites

Sl. No	Composite	Fiber length (mm)	Particulate content (%)	Flexural strength (MPa)
1	Cured epoxy	-	-	20.80
2	Rice husk-epoxy	-	40	30.50
3	Coir-epoxy	30	25	33.85
4	Sisal-epoxy	40	30	45.20
5	CG fiber-epoxy	20	25	145.00

Table 5.Comparison of impact energy of natural fiber/particulate composites

Sl. No	Composite	Fiber length (mm)	Particulate content (%)	Impact Energy (KJ/m^2)
1	Cured epoxy resin	-	-	12.60
2	Rice husk-epoxy	-	30	18.50
3	Coir-epoxy	30	25	23.84
4	Sisal-epoxy	40	30	41.30
5	CG fiber-epoxy	30	25	16.01

Table 6. Comparison of hardness, water absorption and specific wear properties of natural fiber/particulate composites

Sl. No	Composite	Hardness (HV)	Water Absorption (%)	Specific wear rate (mm^3/Nm)
1	Cured epoxy resin	12.5	5.2	0.15
2	Rice husk-epoxy	18.9	4.8	0.10
3	Coir-epoxy	22.5	3.5	0.08
4	Sisal-epoxy	26.8	2.9	0.05
5	CG fiber-epoxy	29.5	1.8	0.01

5. Conclusions

The tensile, flexural, impact, hardness, water absorption and specific wear rate properties of CG fiber – epoxy composites were investigated in the present work .The amorphous nature of resin and semi crystalline nature of fiber were identified using XRD pattern. The presence of hydroxyl group, carbon and oxygen bonding (C=O) and carbon–carbon bonding (C-C) were identified using FTIR spectrum. The weight loss due to temperature and melting point of the material were studied using TGA/DSC plots. The maximum value of tensile strength, flexural strength, impact energy and hardness of 64.61 MPa, 145 MPa, 15.60 kJ/m^2 and 29.5 HV respectively were obtained in randomly oriented short CG fiber reinforced epoxy composites. The minimum value of water absorption of 1.8 % and specific wear rate properties of 0.010 mm^3/Nm were also obtained.

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