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Physical, Mechanical and Microscopic Characterization of Eichhornia Crassipes Fibers (ECF) Reinforced Epoxy Composite using Al_2O_3 as Filler

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Abstract: *The present study investigated the feasibility of utilizing the Eichhornia Crassipes Fiber (ECF) for characterizing and testing the suitability for preparing high strength composite materials. Physical and chemical analysis results revealed that Eichhornia Crassipes Fiber (ECF) has comparatively lower density (1350 kg/m^3) and higher cellulose content (59.86 wt.%)..Physical and chemical analysis results revealed that Eichhornia Crassipes Fiber (ECF) has comparatively lower density (1350 kg/m^3) and higher cellulose content (59.86 wt.%). The chemical functional groups and relevant chemical composition of the ECF were evidenced by the FTIR. The XRD results acknowledged that ECF has the crystallinity index (44.32%) and crystalline size of 5.09 nm respectively. Higher cellulose (59.86 wt.%) and low hemicellulose (9.65 wt.%) content existing in the raw ECF was confirmed by FTIR and chemical analysis. The chemical composition assessment of the untreated and alkali treated ECF confirmed that 5% (w/v) KOH treated for 60 min created positive modifications than other soaking periods (30, 90, and 120 min).The tensile, flexural, and impact strength values of 20 wt.% ECF-reinforced epoxy composites were higher than those of the composite with other quantities of fiber (5, 10,15, and 25 wt.%). The optimally alkalized ECF-reinforced composites had better properties than composites with same weight .The results obtained for the various tests conducted on the KOH solution, Benzoyl Chloride solution, treated fiber samples confirmed the improvement in the physical, mechanical, crystalline and thermal stability properties. Percentage of untreated ECF. Composite specimens were fabricated using the treated fiber samples separately with different fiber loading conditions like 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.% and 25 wt.%. The composites prepared with alkali treated ECFs were found to exhibit better mechanical properties over others; hence they were suggested to be considered for possible structural applications as better alternative material.*

Keywords: *Eichhornia Crassipes Fibers (ECF),Epoxy, Al_2O_3, Alkali , Benzoyl Peroxide, Potassium Permanganate, Stearic Acid, FTIR,NMR,SEM, AFM,TGA & XRD analysis, Ultimate tensile strength, Impact Test, Flexural Test, Tensile Test etc.*

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I. Introduction:

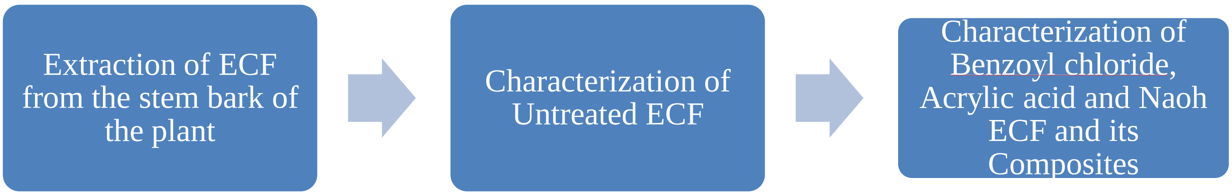
Because of the need for lightweight and robust materials, fibre reinforced polymer composite materials have been developed. To reduce the amount of disposed plastics , renewable and biodegradable natural fibres are incorporated into the polymer matrix which makes the composite a cost effective, light weight having good thermal resistance and wear properties, which expand the application of the natural fibre reinforced composites in diverse fields. Composites are made for specific purposes that require unique mechanical and physical qualities. Mechanical benefits (tensile, flexural, impact) and tribological advantages distinguish composite materials from conventional materials (wear, corrosion and fatigue). A fiber is characterized by its length being much greater compared to its cross sectional dimensions. The dimensions of the reinforcement determine its capability of contributing its properties to the composite. A very large number of polymeric materials, both thermosetting and thermoplastic, are used as matrix materials for the composites. Usually the resinous binders (polymer matrices) are selected on the basis of adhesive strength, fatigue resistance, heat resistance, chemical and moisture resistance etc. The resin must have mechanical strength commensurate with that of the reinforcement. The resin matrix must be capable of wetting and penetrating into the bundles of fibers which provide the reinforcement, replacing the dead air spaces therein and offering those physical characteristics capable of enhancing the performance of fibers. Natural fibers are those that are not synthetic or manmade, but are slender threads created by nature. They are sourced from plants or animals. The use of natural fibers from both renewable and non-renewable resource, such as oil palm, Palmyra, flax and jute to produce composite materials, gained considerable attention in the last few years. Synthetic fibers are extensively used in many applications, but due to high cost, they are slowly replaced by natural fibers. The sources of natural fibres includes plants or animals. The use of plant based natural fibres to produce composite materials, gained significant attention in the last few decades,. The plants, that were observed producing cellulose fibres can be classified into bast fibres (jute, flax, ramie, hemp, and kenaf), seed fibres (cotton, coir, and kapok), leaf fibres (sisal, pineapple, banana and abaca), grass and reed fibres (rice, corn, and wheat), and core fibres (hemp, kenaf, and jute) as well as all other kinds (wood and roots). The natural fibre is used as reinforcement incomposites due to the following advantages,1. Relatively cheap,2. Light weight,3. Reasonable strength,4. High specific modulus,5. Renewable and bio degradable,6. Eco friendly

II. Materials and Methods:

Manna S. et al. were studied alkali treatment to improve physical, mechanical and chemical properties of lignocellulosic natural fibres for use in various applications[1]. Xue Li et al. studied chemical Treatment of Natural Fibres for use in natural fibre-reinforced composite. It was concluded that Chemical treatments can raise the interfacial bonding between the fibre and matrix, and reduce the water absorption characteristics of fibres. Therefore, chemical treatments can be considered in modifying the properties of natural fibres [2]. Biswas et al. concluded that the hardness is decreasing with the increase in fibre length up to 20 mm. An analysis on pulp fibre reinforced thermoplastic composite shows that while the stiffness is increased, the strength of the composite is increased relative to the virgin polymer [3]. Basantakumar Palai et al were investigation, silver nano-particles (AgNPs) were coated on the surface of the Eichhornia Crassipes Fiber (ECF) by single-step hydrothermal method. The coating of AgNPs on the external layer of the ECFs was confirmed through the TGA, SEM, EDX and XRD analysis. The results of thermo gravimetric analysis revealed that thermal stability and maximum degradation temperature of the mM AgNO3 treated ECF was higher than the raw, NaOH treated and other AgNO3 (2 mM, 4 mM, 8mM and 10 mM) treated ECF. Scanning electron scope and energy dispersive X-ray spectroscopy analysis proved the presence of AgNPs on the surface of the fiber with the average particle size of 93 nm. FTIR analysis demonstrated the involvement of OH groups of ECF in AgNPs generation process. XRD analysis displayed the corresponding peaks of silver nanopar-ticles (AgNPs) and silver oxide nano-particles (Ag2ONPs) in the 6 mM AgNO3 treated ECF. Antibacterial test outcomes proved the antibacterial nature of AgNPs coated ECF. All the above findings concluded that AgNPs coated ECF were suitable reinforcement in bio-films used for the medical and food packaging [4]. Gowda et al. studied the mechanical characterization of jute fabricreinforced polyester composites and concluded that jute fibre-based composites show better strengths than the wood-based composites [5]. Raghuram Pradhan et al. studied experimental investigation mainly focused about tensile strength as well as tensile modulus of Fiber reinforced polyester by using Chicken feather fiber. For testing completed with different combination of chicken feather fiber mixed with neat polyester in three different weight percentages. These three different percentages of weight combinations created with four various fiber lengths in "mm". In each combination specimens tests were conducted to identify the tensile strength and tensile modulus values for comparisons of specimens to identify the best combination [6]. Luo and Netravali studied the tensile and flexural properties of polymer composites with different pineapple fibre content and compared them with the virgin resin [7].Sujin et al. studied the effects of aspect ratio and loading on the mechanical properties of Prosopis juliflora fibre-reinforced phenol formaldehyde composites in which mechanical properties were maximum at 23.54% of fibre loading and then decreased. In order to be a suitable reinforcement material, natural fibre must increase the tensile properties of the resin matrix and exceed the critical fibre weight percentage or volume fraction and there must be optimum bonding between the natural fibres and resin matrix .[8].Bledz ki et al. studied Physical, chemical and surface properties of wheat husk, rye husk and soft wood as reinforcements in polypropylene matrix in which he discussed strengths of different composites and the feasibility of utilizing of grain by-products such as wheat husk and rye husk as alternative fillers for soft wood fibre as reinforcement in for composites material [9].Hind Abdella et al. have found because of its inexpensive cost, good mechanical qualities, high specific strength, super adhesiveness, and superior heat and solvent resistance, epoxy is very helpful in load bearing applications (aerospace, vehicle, construction, oil, gas, and marine).[10] Satishkumar Noone et.al examined the effects of treated alkali (NaOH) on composite luffa cylindrica fibre. The fiber value of the composite stays stable at 10%. The mechanical properties variance (Tensile, Flexure and Wear Rate) and eventually contrasted with Glass Fiber Reinforced Polymer. Luffa

Cylindrica (LC) is a tropical plant belonging to the family of cucurbitaceous, with a fruit possessing netting like fibrous vascular system. The luffa cylindrica fibers will be treated with alkali (NaOH) for four hours for chemical treatment. These are used as reinforcements, and epoxy resin was used as a matrix to make up the composite specimens by using hand layup process. The composites can be made by using the raw materials luffa cylindrica fibres of 10% (weight fraction) are chopped into 5 mm length and mixed with epoxy resin and hardener of 90% (weight fraction) [11]. Ahmad MA et al.have prepared where hemp/PET hybrid composite epoxy is used with hemp and PET fiber in the form of interwoven. For composite preparation infusion process is being used .In these tensile strength of woven and interwoven composite PET/Hemp composite is increased and Hemp/PET is lowest. Elastic Module P/H is high and H/P is low. So a conclusion can be drawn that combination of P/H composite were higher than neat H/H[12].G Sai Krishnan et al. were investigated the mechanical properties of chemically treated banana and areca fibers as reinforcement for various applications. Compression molding technique was used to prepare the composites. Fiber loading was varied by 5 and 10 volume weight percentages. Both areca and banana fiber were chemically treated with 5 wt.% sodium carbonate. Tensile, flexural and hardness Testes were carried out. On seeing the results, it was witnessed that the mechanical properties and thermal stability got increased with fiber loading and sodium carbonate treatment. Ten fibers reinforced composite provided the best set of mechanical and thermal properties, while banana fiber reinforced polypropylene composites had slightly better properties as compared to jute fiber reinforced polypropylene composites. [13].N Nallusamy et al. showed that nano Aluminium oxide fillers are incorporated in different weight ratios in the fibre reinforced plastics and the mechanical properties were evaluated. The results showed that tensile strength, flexural strength and shear strength of the glass fibre reinforced plastic improved with addition of nano Al_2O_3 filler particles results in enhancement in Mechanical Properties[14].Basanta Kumar Palai et al.were investigated physical, chemical, structural, crystallographic, thermal and surface topographical properties of fiber extracted from the steam of C(EC) plant was characterized for the first time in this study. Physical and chemical analysis results revealed that Eichhornia crassipes Fiber (ECF) has comparatively lower density (1350 kg/m3) and higher cellulose content (59.86 wt.%). The chemical functional groups and relevant chemical com- position of the ECF were documented by the FTIR. The XRD results acknowledged that ECF has the crystallinity index (44.32%) and crystalline size of 5.09 nm, respectively. Thermogravimetric analysis (TGA) and differential scanning calorimeter analysis (DSC) results of ECF exhibited that it can be thermally stable up to 200° C. All the results of ECF establish that it is a suitable substitute for the artificial fibers used as reinforcement in fiber-reinforced plastics[15].Gassan et al. were tried to improve the mechanical properties of natural fiber by the use of a NaOH treatment processes, they found the improvement in the dynamic modulus of the composite as a result of the use of treated fibers.[16]

Figure 2.1. Images of (a) EC plant, (b) harvested steam, (c) water retting process, (d) extracted fibers



Fourier Transform Infrared Spectroscopy Analysis

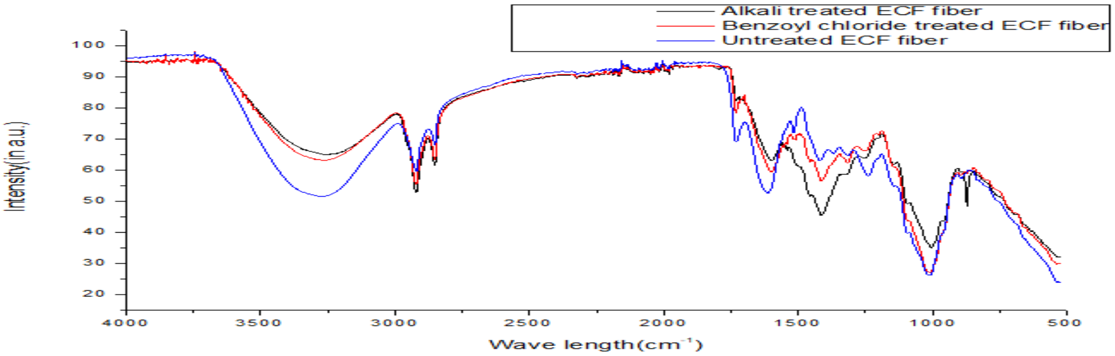


Figure 2.2 shown infrared spectra of the untreated and optimally alkalized ECF.

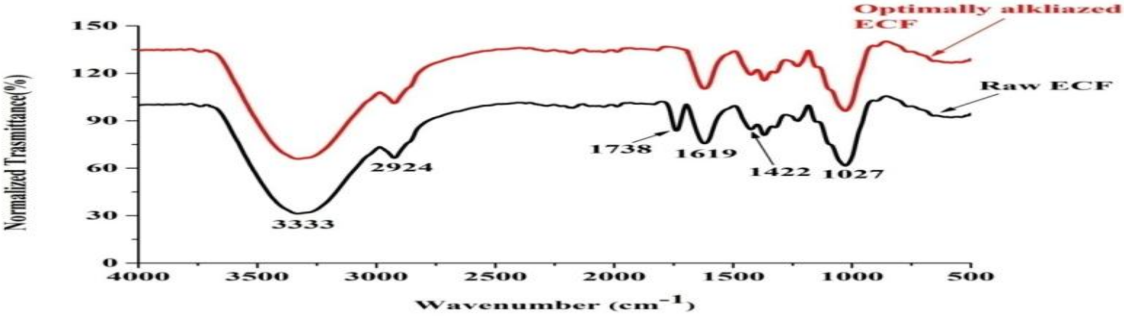
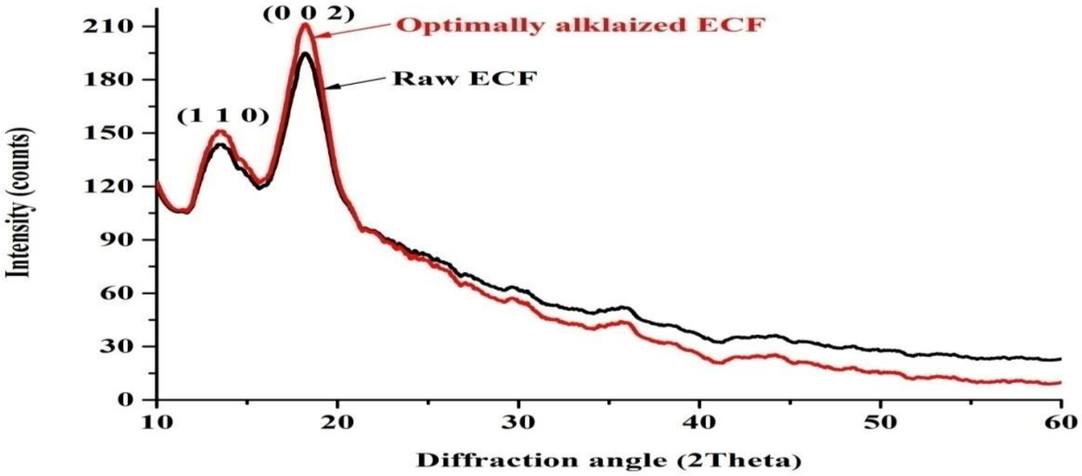
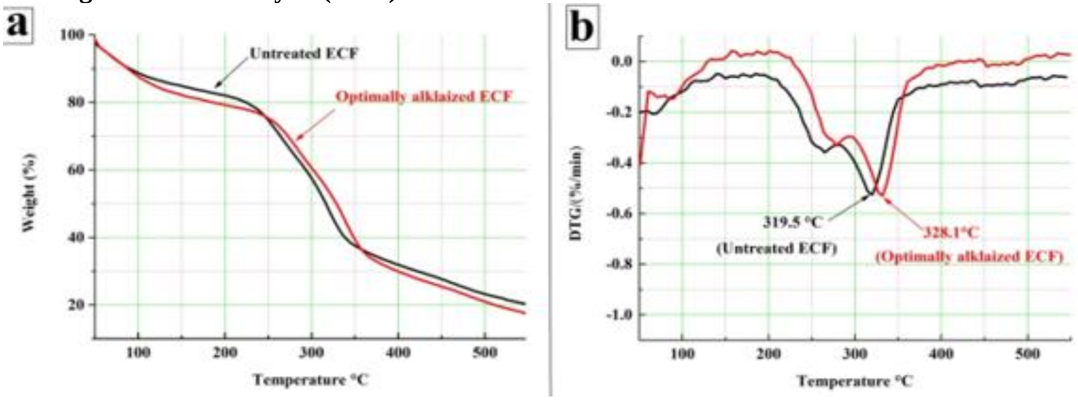


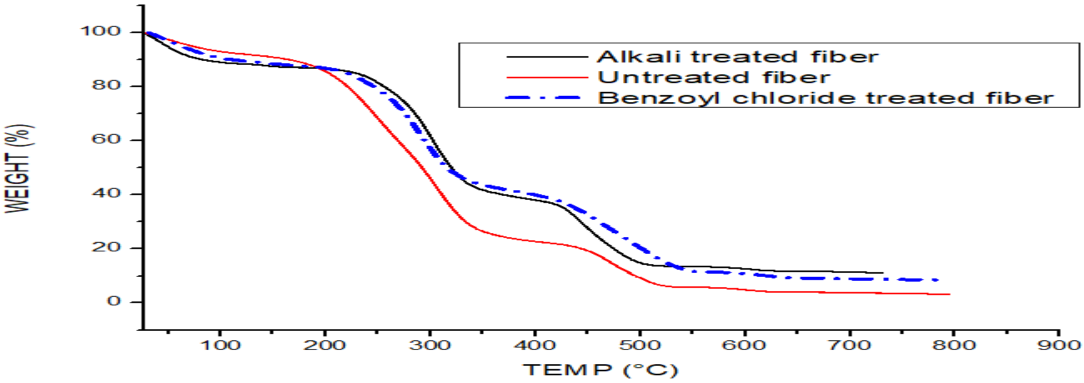
Figure 2.3. Fourier transforms infrared spectrums of untreated and optimally alkalized ECF

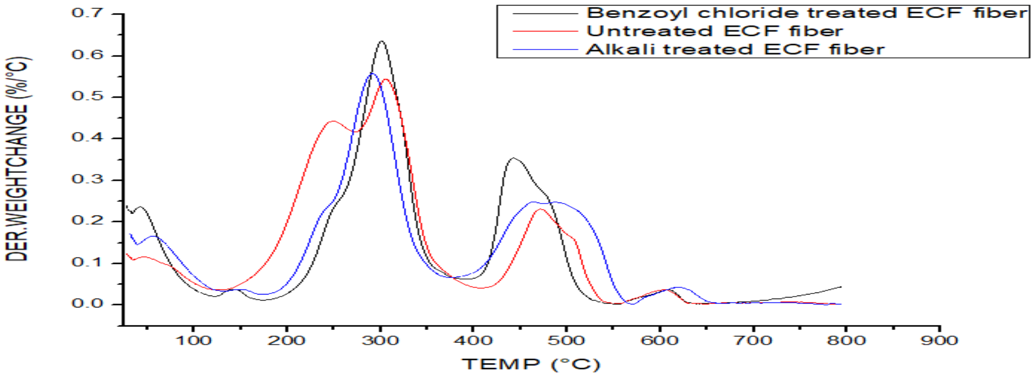
2.1 X-ray diffraction (XRD) analysis



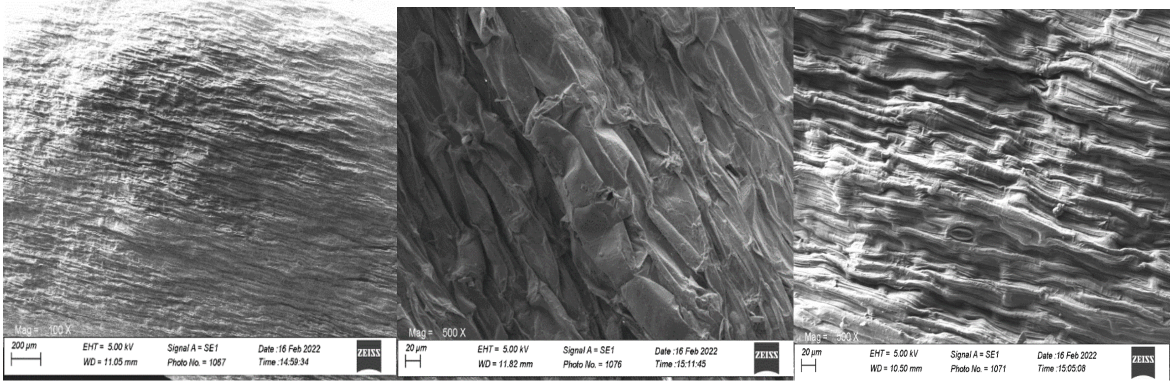
2.2 Thermogravimetric Analysis (TGA)





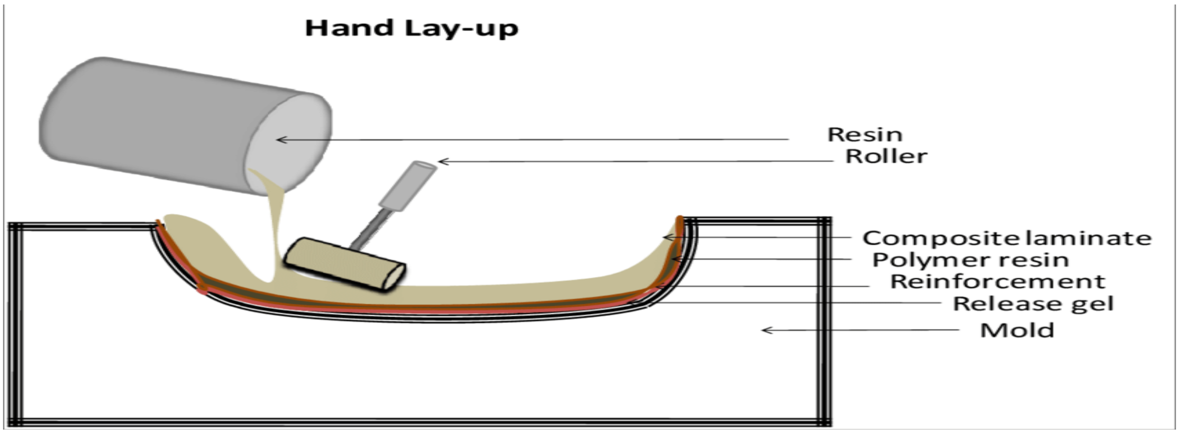


2.3 Scanning Electron Microscope (SEM) Analysis



2.4 FABRICATION AND CHARACTERISATION OF COMPOSITE SPECIMENS

Untreated, optimally alkali-treated, Benzoyl Peroxide treated, Potassium permanganate treated and Stearic acid treated ECF reinforced composites are produced by varying fiber type and fiber weight fractions as 5 wt. %, 10 wt.%, 15 wt.%, 20 wt.% and 25 wt.%. The matrix medium was chosen as epoxy resin. Hand layup method was used for fabricating the proposed composite specimens. The pictorial representation of the fabrication setup is shown in figure 3.6. The dimensions of the die used for the fabrication process were noted to be 300 mm × 300 mm × 3 mm. For the easy removal of the specimens from the die, a clean polythene paper was placed inside the die before filling the fiber and resin. The complete fabrication process was performed at room temperature for over 24 hours.



III. Results and Discussions:

Mechanical Property Analysis on the Composite Specimens

Tensile Test

Specimens were prepared according to the ASTM standard D3039-17 for carrying out the tensile strength test. This standard has been revised by the ASTM International, West Conshohocken on 2017. The experimental analysis was performed for all the twenty five composite specimens on a Deepak Poly Plast: DTRX model Universal Testing Machine (UTM) whose load capacity was noted as 250 KN

Flexural Test

The flexural strength values of the prepared specimens were tested using three-point bend test on a DTRX model Universal Testing Machine with 250 KN load capacity. Specimens were prepared with ASTM standard D790-15 for the flexural strength test (Arthanrieswaran et al. 2016). This standard has been revised by the ASTM International, West Conshohocken on 2015.

Impact Test

Izod impact test was performed on the specimens to predict the impact strength of the proposed composite materials onDeepak Poly Plast digital impact strength tester. The impact test specimens were prepared in compliance with the ASTM standard D256-10. This standard has been revised by the ASTM International, West Conshohocken on 2018. The capacity of the tester was noted to be 0.5 to 50 J energy (Arthanrieswaran et al. 2016).

SEM Analysis

After the tensile testing, broken specimens of 20 wt.% untreated and chemical treated fiber reinforced composites were inspected by using a VEGA3 scanning electron microscope. The images were recorded with the settings of 5 kV accelerating voltage and 500X magnification.

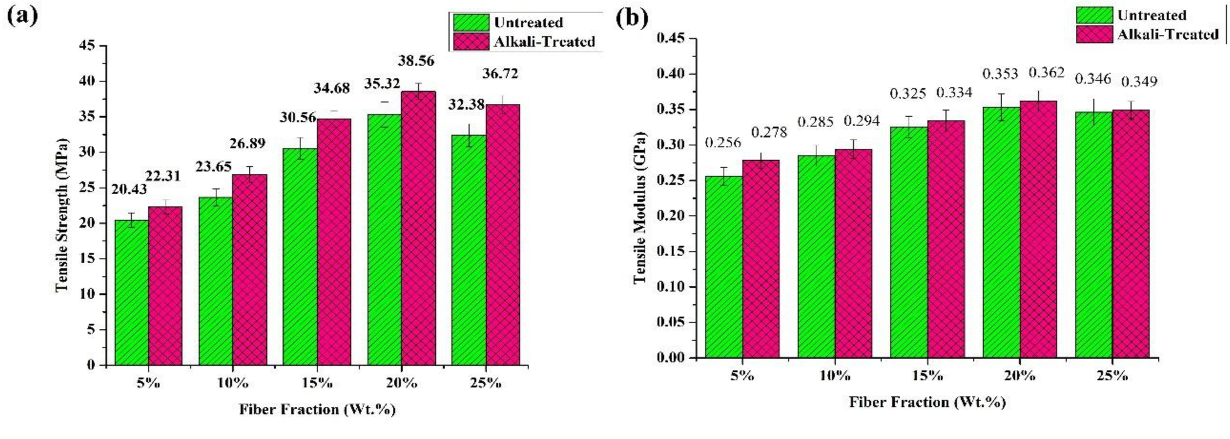


Figure 3.1. (a) Ultimate tensile strength and (b) tensile modulus of untreated and optimally alkalized ECF reinforced epoxy composites

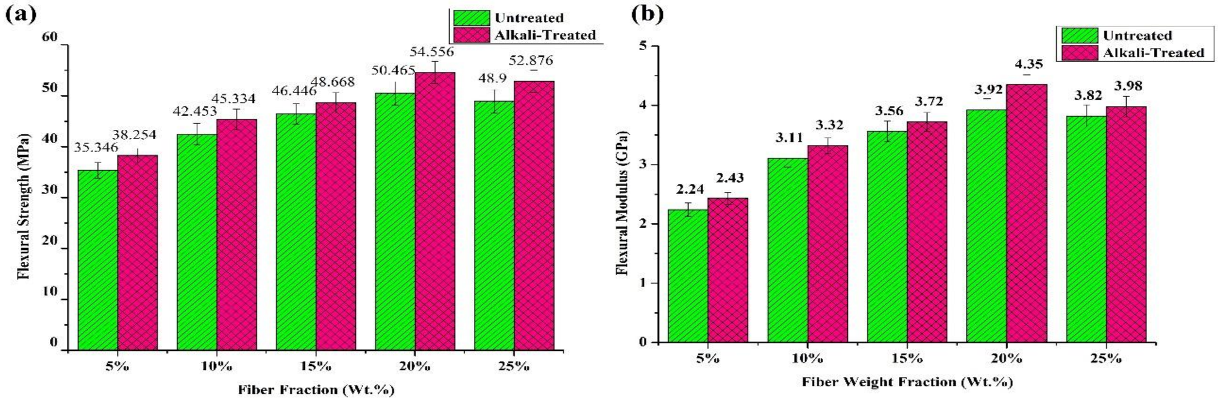


Figure 3.2. (a) Ultimate flexural strength and (b) flexural modulus of untreated and optimally alkalized ECF reinforced epoxy composites

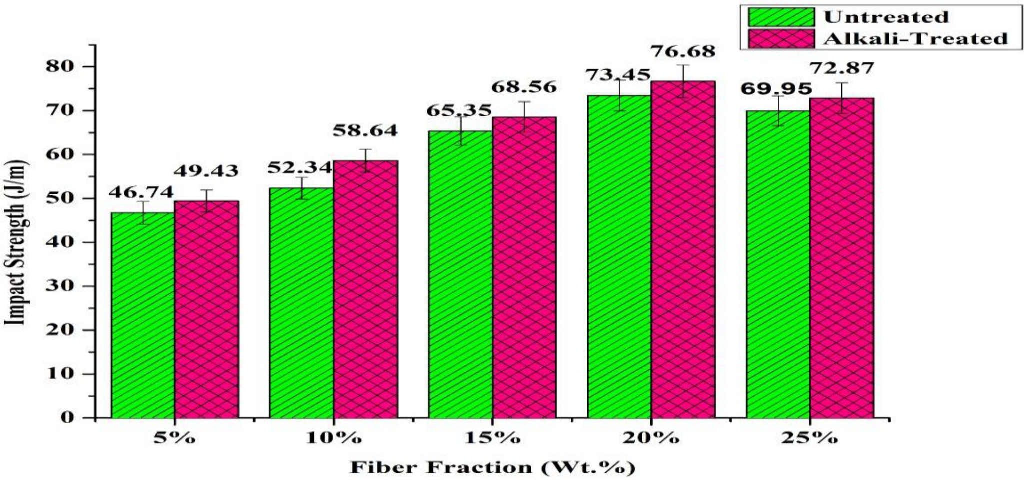


Figure 3.3. Impact strength of untreated and optimally alkalized ECF reinforced epoxy composites

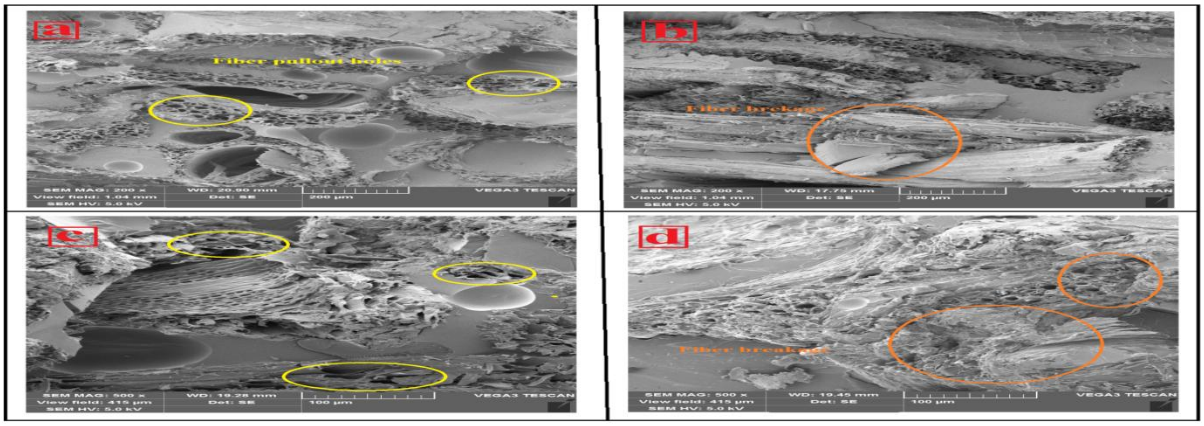


Figure 3.4. SEM images of tensile fractured surfaces of 20Wt. % (a, c) untreated and (c, d) optimally alkalized ECF reinforced composites

IV. Conclusions:

The present study investigated the feasibility of utilizing Eichhornia Crassipes Fiber (ECF) for the possible structural applications. For that, the untreated, Alkali treated, Benzoyl peroxide treated, Potassium permanganate treated, Stearic acid treated, Silane treated ECF samples were subjected to several experimentations for characterizing them. Silver nano particles were coated on the ECFs in order to check them for antibacterial application. Composite specimens were prepared with the untreated and treated ECFs. Mechanical characterization study was prepared on the prepared composite samples. Brake pads were prepared using the untreated, alkali treated and silane treated ECFs; and the suitability analyses were performed and investigated. The conclusions made from the results carried out during the entire work are presented below.

- Higher cellulose (59.86 wt.%) and low hemicellulose (9.65 wt.%)content existing in the raw ECF was confirmed by FTIR and chemical analysis. Low density (1350 kg/m^3) and higher crystallinity index (44.32%) were favorably found with the raw ECF.
- The higher average surface roughness (101.84 nm) and positive roughness skewness (0.213 nm) values of ECF was evident by the AFM.
- The chemical composition assessment of the untreated and alkali treated ECF confirmed that 5% (w/v) NaOH treated for 60 min created positive modifications than other soaking periods (30, 90, and 120 min).
- The excess hemicellulose fraction in the optimally alkalized ECF was eliminated, which was verified by the FTIR and NMR analyses.
- The increase in the CI (44.32% to 47.61%) value of the alkalized ECF was evidenced by the XRD.
- Surface topographical modifications on the optimally alkalized ECF were documented by the AFM analysis.
- The tensile, flexural, and impact strength values of 20 wt.% ECF-reinforced epoxy composites were higher than those of the composite with other quantities of fiber (5, 10, 15, and 25 wt.%). The optimally alkalized ECF-reinforced composites had better properties than composites with same weight percentage of untreated ECF.
- The results obtained for the various tests conducted on the NaOH solution, Benzoyl peroxide solution, Potassium permanganate solution and Stearic acid solution treated fiber samples confirmed the improvement in the physical, mechanical, crystalline and thermal stability properties.Comparatively, the results of stearic acid treated fiber samples were found to be better.
- Composite specimens were fabricated using the treated fiber samples separately with different fiber loading conditions like 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.% and 25 wt.%.The composites prepared with stearic acid treated ECFs were found to exhibit better mechanical properties over others; hence they were suggested to be considered for possible structural applications as better alternative material.
- Acoustic property study TGA analysis proved that 6mM $AgNO_3$ treatment has improved thermal stability than the raw, NaOH treated and other $AgNO_3$ (2mM, 4mM, 8mM and 10mM) treated ECF.
- Scanning electron microscopic analysis and EDX analysis confirmed the deposition of silver nano-particles (AgNPs) on surface of ECF.
- Antibacterial test results proved that AgNPs coated ECF exhibited excellent antibacterial activity against Escherichia coli, Staphylococcus epidermidis and Bacillus subtilis bacteria.
- Silane treated Eichhornia Crassipes Fibers had increased cellulose content of 72.64% with an enhanced density of 1449 kg/m^3 because of elimination of amorphous contents.
- Brake pads were fabricated as per the automotive standards by using untreated, alkali treated and silane treated Eichhornia crassipesfibers.Silane treated Eichhornia Crassipes fibers based brake pads exhibited enhanced shear strength, hardness, low porosity, acetone extraction, loss on ignition.
- Silane treated Eichhornia Crassipes fibers based brake pads showed a better coefficient of friction under both fade and recovery conditions with good wear resistance. This was due to thermal stability of treated fibers and better bonding of fiber with the matrix leading to increased hardness. Alkali treated Eichhornia Crassipes Fibers based brake pads showed intermittent results.
- Silane treated Eichhornia Crassipes Fibers based brake pads displayed adequate sized contact plateaus with less back transfer patches due to better bonding of fiber with resin.

V. Scope for Future Work

The natural fiber addressed in this work has been characterized and reported. The improvement in the various desirable properties of natural fiber has been analyzed by subjecting to various chemical treatments and documented. Composite specimens have also been prepared and their suitability for antibacterial application and brake pad application has been investigated. However, this work can be further extended in the following ways.

i. Industrial products can be developed using the proposed biodegradable Eichhornia Crassipes Fibers.
ii. ECFs can be reinforced with other types of thermosetting and thermoplastic matrix media to fabricate and test the composites.
iii. Unidirectional and bidirectional mats can be prepared using the ECFs and their composites can be investigated for the mechanical characteristics.
iv. ECFs can be hybridized with other natural fibers to study the improvement in required properties.
v. ECFs can be hybridized with synthetic fibers and high strength composite structures can be developed.
vi. Further, there is a large scope for Eichhornia Crassipes Fibers for future study.
   a) Dynamic study, b) Water absorption study, c) Energy absorption study

b) Acoustic property study

References:

[1]. Manna S., Saha P, S. Chowdhury, S. Thomas, and V. Sharma. 2017, **“** Alkali treatment to improve physical, mechanical and chemical properties of lignocellulosic natural fibres for use in various applications”. Lignocellulosic Biomass *Prod Ind.,* 47–63

[2]. Xu Li, Lope G. Tabi, **“** Chemical Treatment of Natural Fibres for use in natural fibre-reinforced composite”, Springer Science and Business Media, 2007.

[3]. Biswas S., Kindo S., PatnaikaEffect of Length on Coir Fibre Reinforced Epoxy Composites”, Fibre and Polymers 12, pp. 73-78.

[4]. Basantakumar Palai, Saroj Kumar Sarangi, and Siba Shankar Mohapatra, “ Characterization of Silver Nano-particle Coated Eichhornia Crassipes Fiber for Antibacterial Application”,JOURNAL OF NATURAL FIBERS,2020 Taylor and Francis group, https://doi.org/10.1080/15440478.2020.1788492.

[5]. Gowda, T. M., Naidu, A. C. B. & Chhaya, R, **“** Some Mechanical Properties of Untreated Jute Fabric-Reinforced Polyester Composites”, Composites Part A: Applied Science and Manufacturing, Vol.30(3), pp. 277-284.1999.

[6]. Raghuram Pradhan, K Muralidharan, AK Saravanan, M Raajasekar, “ Experimental investigation of tensile strength and tensile modulus of fiber reinforced polyester by using chicken feather fiber”,AIP Conference Proceedings, 2020, Volume-2283, Issue-1.

[7]. Luo, S. & Netravali, A. N, **“** Mechanical and thermal properties of environmentally friendly green composites made from pineapple leaf fibres and poly (hydroxybutyrate-covalerate) resin**”**,Polymer Composites,Vol. 20(3), pp. 367-378.1999.

[8]. Sujin Jose A, Athijayamani A, Ramanathan K, Sidhardhan S**“** Effects of Aspect Ratio and Loading on the Mechanical Properties of Prosopis julifloraFibre-reinforced Phenol Formaldehyde Composites**”**, FIBRES & TEXTILES in Eastern Europe; 25, 4(124): 59-64. DOI: 10.5604/01.3001.0010.2664.2017.

[9]. Bledzki A. K., Mamun A. A., Volk J.,Physical, **“** chemical and surface properties of wheat husk, rye husk and soft wood and their polypropylene composites**”**, Elsevier. Composites part A (41) 2010. Pp 480-488.

[10]. Hind AbdellaOui, Abou elkaeem Qaiss, **“** Investigation of deformation behaviour of epoxy based composites of Failure analysis in Biocomposites**”**, Fibre composites and hybrid composites,2019.

[11]. Satishkumar Noone, K Purushothaman, Raghuram Pradhan, “ An investigation on luffa cylindrica fiber reinforced epoxy composite”, Materials Today-Elsevier, Volume 33,Pages1026-1031

[12]. Ahmad MA, Majid MS, Ridzuan MJ, Firdaus AZ, Amin NA**“** Tensile properties of interwoven hemp/PET (Polyethylene Terephthalate) Epoxy composites**”**. ,In Journal of Physics: Conf Series. 2017; 908:1742-6596.

[13]. G Sai Krishnan, Raghuram Pradhan, Ganesh Babu Loganathan, “ Investigation on Mechanical Properties of Chemically Treated Banana and Areca Fiber Reinforced Polypropylene Composites”, Advances in Lightweight Materials and Structures, Springer, Singapore, Pages 273-280.

[14]. N Nallusamy, Dr M Suresh, Dr Sundarajan Rajkumar and AK Lakshminarayanan, GSheshanandan, Dhandapani Ravindran, T Sornakumar, **“** Effect of NanoAluminium Oxide fillers on the properties of FRP polymer Matrix composites**”**,Applied Mechanics and Materials. 2012.

[15]. Basantakumar Palai, Saroj Kumar Sarangi, and Siba Shankar Mohapatra,“ Investigation of Physiochemical and Thermal Properties of Eichhornia Crassipes Fibers”, JOURNAL OF NATURAL FIBERS,2020 Taylor and Francis group, https://doi.org/10.1080/15440478.2019.1691110.

[16]. J. Gassan, Jochen and A. K. Bledzki, “Possibilities for improving the mechanical properties of jute/epoxy composites by alkali treatment of fibres”, Composites Science and Technology, vol. 59, no.9, pp. 1303–1309, 1999.

Rabindra Kumar Panigrahi, et. al. “Physical, Mechanical and Microscopic Characterization of Eichhornia Crassipes Fibers (ECF) Reinforced Epoxy Composite using Al2O3 as Filler”. *IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE),* 19(4), 2022, pp. 06-14.