

MECHANICAL PROPERTIES OF SURFACE MODIFIED AND RAW NATURAL FIBER FOR COMPOSITE PREPARATION

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Engineering degree in Mechanical engineering.

By

MUPPARAJU SUDHEER (37150124)
NIKHIL KUMAR REDDY KUMMETHA (37150130)



**DEPARTMENT OF MECHANICAL ENGINEERING
SCHOOL OF MECHANICAL ENGINEERING**

SATHYABAMA
INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119

Accredited with Grade "A" by NAAC

MARCH 2021



SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)



Accredited with “A” grade by NAAC

Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai - 600 119

www.sathyabama.ac.in

DEPARTMENT OF MECHANICAL ENGINEERING

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of MUPPARAJU SUDHEER (37150124) who carried out the project entitled **“MECHANICAL PROPERTIES OF SURFACE MODIFIED AND RAW NATURAL FIBER FOR COMPOSITE PREPARATION”** under my supervision from December 2020 to April 2021.

Internal Guide

Mr. R. SIVA, M.E., (Ph.D)

Head of the Department

Dr. G. ARUNKUMAR, M.E., (Ph.D)

Submitted for Viva voce Examination held on _____

Internal Examiner

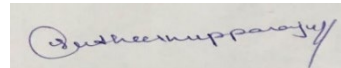
External Examiner

DECLARATION

I MUPPARAJU SUDHEER (37150124) hereby declare that the Project Report entitled **“MECHANICAL PROPERTIES OF SURFACE MODIFIED AND RAW NATURAL FIBER FOR COMPOSITE PREPARATION”** done by me under the guidance of **Mr. R. SIVA, M.E., (Ph.D)** is submitted in partial fulfillment of the requirements for the award of Bachelor of Engineering degree in Mechanical engineering.

DATE: 03-04-2021

1.

A rectangular box containing a handwritten signature in blue ink. The signature appears to be 'Sudheer Mupparaju'.

PLACE: Chennai

SIGNATURE OF THE CANDIDATE

ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to Board of Management of **SATHYABAMA** for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to **Dr. S. PRAKASH, M.E., Ph.D**, Dean, School of Mechanical Engineering and **Dr. G. ARUNKUMAR, M.E., Ph.D**, Head of the Department, Department of Mechanical engineering for providing me necessary support and details at the right time during the progressive reviews.

I would like to express my sincere and deep sense of gratitude to my Project Guide **Mr. R. SIVA, M.E., (Ph.D)**, for his valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

I would like to express heartfelt thanks to **Mr. NIVIN JOY THYKATTUSSERRY, M.E., (Ph.D.)**, Project coordinator, Department of Mechanical Engineering for kindly assisting, intimation of the necessary information and corrections on the right time.

Further, I wish to express my thanks to all Teaching and Non-teaching staff members of the Department of Mechanical engineering who were helpful in many ways for the completion of the project.

ABSTRACT

Depending upon the current scenario, there is an increased awareness that non-renewable resources are becoming scary, and dependence on renewable resources is growing. The 21st century may be the cellulosic century as natural fiber is replacing traditional materials due to its primary advantage of being lightweight. The substituted natural fibers play an important role in composite manufacturing due to their superior environmental benefits. Embracing the benefits this research has been done on chemical treated and raw extracted *coccinia grandis* fiber. The tensile test is done on individual fiber sample with incremental improvement of gauge length 10mm, up to 50mm, volume of 30 samples each. The gauge length that exhibits higher tensile strength is carried out for chemical treatment with sodium carbonate (Na_2CO_3) of 5%, 10% and 15%. After tensile testing of the treated fiber, a higher result is considered. Mechanical properties are compared between fibers to illustrate the enhancements of alkali treated over untreated fiber to perform chemical analysis. The outcome of the chemical analysis implies that, after alkali treatment, the cellulose, density and ash content of CGF went whereas hemicelluloses lignin, moisture, pectin and wax contents depreciated. X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray spectroscopy (EDX), Fourier-Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA) are performed and compared between Raw and treated fiber to find crystalline size, surface roughness, functional groups and thermal stability of fiber respectively. Natural fiber is very much capable of replacing the synthetic fiber because of the ongoing phenomenon of climate changes.

LIST OF CONTENTS

CHAPTER No	TITLE	PAGE No
	ABSTRACT	v
	LIST OF FIGURES	ix
	LIST OF TABLES	xi
	LIST OF SYMBOLS AND ABBREVIATIONS	xii
1	INTRODUCTION	1
	1.1 NATURAL FIBER	1
	1.2 FIBER TYPES	1
	1.3 BAST FIBERS	2
	1.4 COCCINIA GRANDIS	2
	1.5 XRD (X-RAY DIFFRACTION)	3
	1.6 TGA (THERMOGRAVIMETRIC ANALYSIS)	4
	1.7 FESEM (FIELD EMISSION SCANNING ELECTRON MICROSCOPY)	5
	1.8 EDAX (ENERGY DISPERSIVE X-RAY ANALYSIS)	6
	1.9 FTIR (FOURIER TRANSFORM INFRARED SPECTROSCOPY)	6
2	LITERATURE SURVEY	7
3	AIM AND SCOPE OF THE PROJECT	13

	3.1 AIM OF THE PROJECT	13
	3.2 SCOPE OF THE PROJECT	14
4	MATERIALS AND METHODS	15
	4.1 METHODOLOGY	15
	4.2 MATERIALS REQUIRED	16
	4.3 FIBER EXTRACTION	16
	4.4 PHYSICAL ANALYSIS	17
	4.5 DENSITY MEASUREMENT	17
	4.6 SINGLE FIBER TENSILE TEST	18
	4.7 ALKALI TREATMENT	19
	4.8 CHEMICAL ANALYSIS	21
	4.9 FTIR ANALYSIS	22
	4.10 XRD ANALYSIS	23
	4.11 TGA ANALYSIS	23
	4.12 SEM ANALYSIS	25
5	RESULTS AND DISCUSSION	26
	5.1 CHEMICAL ANALYSIS	26
	5.2 TENSILE STRENGTH	27
	5.2.1 UNTREATED FIBER	27

	5.2.2 TREATED FIBER	29
	5.2.3 DENSITY MEASUREMENT	30
	5.3 SEM ANALYSIS	31
	5.4 FTIR ANALYSIS	32
	5.5 XRD ANALYSIS	33
	5.6 TGA ANALYSIS	35
6	SUMMARY AND CONCLUSION	37
	6.1 SUMMARY	37
	6.2 CONCLUSION	38
	REFERENCES	39
	APPENDIX	45

LIST OF FIGURES

FIGURE No	TITLE	PAGE No
1.1	Flow chart of natural fibers	1
1.2	Classification of TGA	4
4.1	Workflow flow chart	15
4.2	Extraction images; (a)CGF plant;(b) Stem of CGF;(c) Stem immersed in water;(d) Intermediate state of retting process;(e) Wet extracted fiber;(f) Dry fiber	16
4.3	Digital micrometer	17
4.4	Samples for single fiber tensile test	18
4.5	Instron 5500R machine	19
4.6	Alkali treatment process; (a) Chemical weighing; (b) 200ml distilled water; (c) Chemical mixture; (d) Dissolved chemical; (e) Fiber immersed in solution; (f) Fiber immersed for 6hours; (g) Single fiber chemical treatment with different concentrations; (h) Single fiber heat treatment; (f) Chemical analysis heat treatment	20
4.7	Fiber for chemical analysis	21
4.8	FTIR operation machine	22
4.9	XRD operated image	23
4.10	TGA equipment	24
4.11	SEM machine	25
5.1	Untreated CGF tensile strength values comparison	28
5.2	Alkali treated CGF Tensile Strength values Comparison	30

FIGURE No	TITLE	PAGE No
5.3	SEM image of untreated CGF	31
5.4	SEM image of alkali treated CGF	32
5.5	FTIR analysis graph	33
5.6	XRD analysis graph	34
5.7	Untreated CGF TGA	35
5.8	Treated CGF TGA	36

LIST OF TABLES

TABLE No	TITLE	PAGE No
5.1	Chemical analysis results comparison	27
5.2	Mechanical properties of Untreated CGF	28
5.3	Mechanical properties of Alkali treated CGF	30

LIST OF SYMBOLS AND ABBREVIATIONS

CG	<i>Cocinnia Grandis</i>
CGF	<i>Cocinnia Grandis</i> fiber
NFC	Natural-Fibre Composites
PP	Polypropylene
PE	Polyethylene (PE)
PVC	Polyvinyl Chloride (PVC)
CQFS	Cissus quadrangularis stem Fiber
ISO	International organization for standardization
XRD	X-Ray Diffraction
ICDD	International Centre for Diffraction Data
PDF	Powder Diffraction File
TGA	Thermogravimetric Analysis
FESEM	Field Emission Scanning Electron Microscopy
SEM	Scanning electron microscopy
EDAX	Energy Dispersive X-Ray Analysis
FTIR	Fourier Transform Infrared Spectroscopy
NDT	non-destructive testing
. wt%	Weight percentage

CHAPTER-1

INTRODUCTION

1.1 NATURAL FIBER

Natural fiber composites are becoming more common as people become more environmentally conscious, furthermore, due to the material's low cost and density, acceptable specific properties, separation ease, CO₂ neutrality, biodegradability, and energy regeneration. Recyclable properties are an important phenomenon of our generation. The materials are durable, reliable, lightweight, and with superior mechanical properties to those of other materials also preferred in industries, including automobile, aviation, and construction. Fibrous plants are developed or grown with the help of photosynthesis. The term lignocellulosic fibers represent natural fibers, but it refers to fibers containing lignin and cellulose. Based on the use case, natural fibers are divided into two groups, first being primary, stating they are grown from the fiber content of the plant such as kenaf, cotton and etc. other one is secondary grown from those that have fibers as a by-product of another primary use such as pineapple, cereal and etc.

1.2 FIBER TYPES

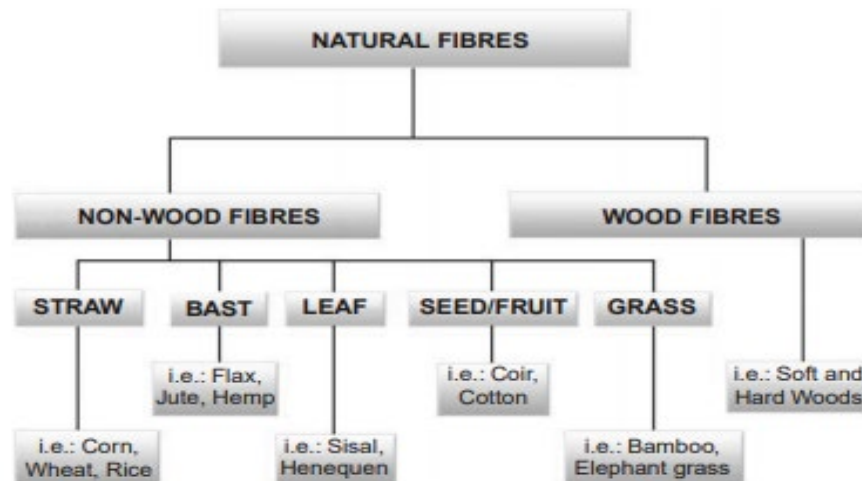


Fig:1.1 : Flowchart of natural fibers

Types of natural fibers along with their core fibers are represented in fig 1.1.

1.3 BAST FIBERS

Fiber extracted from the inner layer of the fibrous plant is called bast fiber. These fibers are underneath a thin bark in the form of fiber bundles or strands that run parallel to the stem's length. Bast strands come in a range of sizes and widths, varying from 1 mm to 100 cm.

1.4 COCCINIA GRANDIS FIBER

This project is carried out with *Coccinia Grandis*, a bast fiber used in biomedical applications for a long time. Synthetic or human-made fibers are widely used in product development, this fiber has the potential to replace them as natural fibers are gaining more awareness in all industries like aviation, construction, and automobile. This fiber is extracted through a water retting process in which the stem is removed and immersed in water for three weeks, during which the fiber is separated from the inner layer of the stem. After that, the extracted fiber is exposed to the sun for three days. Natural fibers being friendly to nature, strong, low cost, and even lightweight, have already started to replace glass and carbon in numerous engineering applications.

The automobile industry has incorporated the advantages gained from natural fibers, and the Natural-Fiber Composites (NFC) made through the combination of natural fibers with different polymers such as Polylactic Acid (PLA), Polypropylene (PP), Polyethylene (PE), and Polyvinyl Chloride (PVC). with the increasing demand, many major industries and manufacturers have invested their resources in developing fiber-reinforced composites. Fiber-reinforced composites have gained importance in the automotive sector, where high mechanical properties and dimensional stability must be coupled with low weight. Natural fibers have their limitations, such as sensitivity to moisture, and their polymer matrices bonding might be weak. Moreover, there are some concerns over natural-fiber consistency, temperature limits, chemical treatment, and quality.

Based on this discrepancy, the research work has been done on *Coccinia Grandis*, we can test the fiber for its best mechanical properties, thermal properties, chemical analysis, and surface morphology of the raw extracted and alkali-treated natural fiber without even making the composite. This process helps in the predicament of best performing gauge length of the fiber composition without even going through all the possible combinations of composite preparation with much effective cost and time. In this report, we have compared the raw extracted fiber with alkali-treated fiber to demonstrate significant changes in all mechanical properties, chemical analysis, thermal properties, and surface morphology.

First, we have taken raw extracted fiber of 30 samples of each gauge length 10mm, 20mm, 30mm, 40mm, and 50mm; the diameter of each fiber is measured at three points with an electron microscope. This fiber has undergone a tensile test individually with a varying load, which exhibited a significantly greater 10mm gauge length. The chemical treatment is performed on the 10mm gauge length fiber with 5%, 10%, and 15% sodium carbonate (Na_2CO_3). The ratio for the composition of chemical and distilled water is 1:20. The results of all the tests that are done have shown the fiber's development by using a new chemical. The fiber is done to know the cellulose, hemicellulose, wax, lignin, moisture, ash, and pectin based on the data chemical analysis of the fiber.

1.5 XRD (X-RAY DIFFRACTION)

Studying the XRD patterns of *Coccinia Grandis* is very advantageous in several applications and is an NDT tool for observing the crystalline structure of materials. XRD analysis classifies the crystalline phases present in a substance and reveals chemical composition details by observing the crystal structure. Application and advantages of XRD include recognizing the various crystalline phases and their orientation in the material, dimensions of the lattice, impact, and scale of fabric.

XRD patterns of *Coccinia Grandis* dealt with the exploration of various additional applications. The accurate way to analyze dimensions of each and every unit cell along with identification of the crystalline material structure, X-ray powder diffraction is

preferred. The method of X-ray diffraction is also widely used to research crystal structures and atomic spacing. A cathode-ray tube produces the X-rays, emits monochromatic radiation, collimated to focus the beam, and guided the piece. The diffracted rays obtained after X-rays are focused on the sample. The most critical part in the process of XRD is angle between the diffracted and incident beams. Beyond that, the instrumentation for powder and single-crystal diffraction differs.

1.6 TGA (THERMOGRAVIMETRIC ANALYSIS)

TGA a thermal analytical method explaining mass changes under a temperature increase regulated by the program. Classification of TGA is represented in fig 1.2. TGA measures the difference in mass loss of weight which includes evaporation, decomposition when the product is in a gaseous state, and even oxidation when the fiber sample is gaining weight. Information obtained on material thermal stability and its proportion of volatile components by controlling the weight shift that occurs when the constant rate of heat is given to the specimen by performing TGA. In a controlled atmosphere, with variations in temperature and time, the mass changes in a material are observed in TGA. The main objective is to measure the heat resistance and composition of a material. TGA is most useful for dehydration, decomposition, desorption, and oxidation processes.

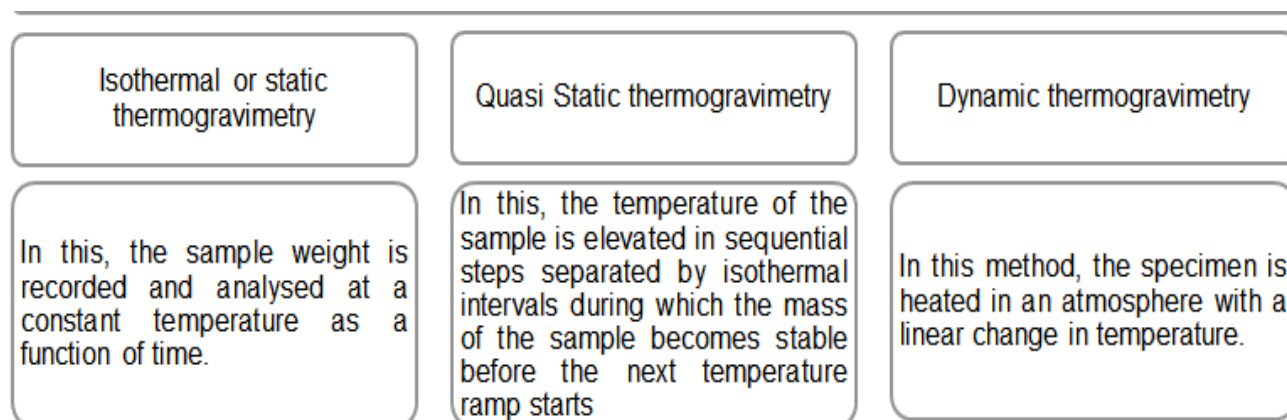


Fig:1.2 : Classification of TGA

TGA is generally conducted in the thermogravimetric analyzer. In a thermogravimetric analyzer by changing the temperature of a sample over time measures mass continuously. The three base measurements in TGA are mass, time, and temperature, other additional data is derived from main base measurements. The thermogravimetric data obtained from a temperature variation is represented in a graph with a mass on the y-axis and temperature on the x-axis. If the TGA graph heads down we can conclude that there might be a weight loss taken place, whereas if the graph heads upward in the restricted temperature limits, we can conclude that weight gain has taken place. The plot, which is often smoothed, will be the TGA curve. Characteristics of polymeric fabrics such as thermoplastics, thermosets, elastomers, composites, synthetic products, textures, coatings, colours, and fuels are being researched by using TGA to study temperature degradation of materials.

1.7 FESEM (FIELD EMISSION SCANNING ELECTRON MICROSCOPY)

FESEM provides terrain, surface morphology, and elemental data with virtually unlimited field of depth. FESEM micrographs visualize very tiny topographic details that produce clear, less distorted images with a spatial resolution better than conventional scanning electron microscopy (SEM). The principle behind the FESEM is narrower test beams at low and high electron energy provided by the electron gun of electron microscope with a cathode present in the electron gun, resulting in improved spatial resolution and minimized sample damage.

The advantages of FESEM are low-kinetic energy which reduces electron penetration into the material surface immediately that produces high-quality, low-voltage images with negligible sample electrical charges, and conducting materials are not needed for insulating materials. FESEM uses include cross-sectional measurements of semiconductor devices for gate widths, gate oxides, film thicknesses, and design details, advanced assessment of chemical thickness, and composition uniformity.

1.8 EDAX (ENERGY DISPERSIVE X-RAY ANALYSIS)

EDAX is an X-ray method also referred to as EDX, used to identify a material's chemical composition. From EDAX we can speculate the chemical composition of the fiber material and even its distribution and concentration. Both quantity and consistency measurement can be determined using EDAX. The results obtained from the EDAX test are by capturing the X-rays emitted by the fiber material when electromagnetic radiation is imposed on it.

The electron beam enters an atom's inner shell, taking out an electron from the shell and leaving an electron-hole that is positively charged, which pulls a negatively charged electron from the outer shell so that the gap is filled. Depending on the electron beam acceleration voltage, the X-rays emitted differ.

1.9 FTIR (FOURIER TRANSFORM INFRARED SPECTROSCOPY)

FTIR analysis is a technique for distinguishing organic, inorganic, and polymeric materials by scanning them with infrared light. A shift in the material composition is indicated by changes in the absorption band pattern. FTIR classifies and describes unknown materials, detect pollutants in a sample, locate additives, and also detect decomposition and oxidation.

FTIR can obtain high spectral resolution data over a numerous range of wavelengths, generally between 5000 and 400 cm^{-1} for the mid-IR region and between 10,000 and 4000 cm^{-1} for the near-IR region. Calculating sample that how of the monochromatic light beam it absorbed is a simple way to do this absorption calculation. Regulating parameters can give us many measurements for each different wavelength. FTIR is a never-ending process that measures many frequencies of light simultaneously this provides a benefit for FTIR with a higher ratio of signal to noise in a given scan period.

CHAPTER-2

LITERATURE SURVEY

Garette Jebadurai.S, et.al., (2018) From the reference from this paper we selected new fiber *coccinia grandis*. As the research work because a lot of research is going on natural fiber due to its benefits. Jute, hemp, coir, banana, kenaf, and other fibers have a lot of observations and findings when it comes to composite preparation, so the new fiber *Coccinia Grandis* was chosen because of its high cellulose content, low density, and thermal stability. Various tests are performed on fiber to determine mechanical, thermal, anatomical, and Physico-chemical properties of CGF, that prove this fiber is fit to replace the harmful synthetic fiber. Surface modification is carried out using various chemicals to assess the effects of alkaline treatment on fiber to strengthen the fiber. The properties of various fibers are compared, such as cellulose, hemicellulose, wax, lignin, ash, tensile properties, physical properties, and so on. As can be seen from this journal, this analogy was critical in recognizing the properties of natural fiber.

Senthamaraikannan.P, et.al., (2018) The simple method to extract the fiber using the knitting process is discussed in this research work. The CGF stem is collected and placed in water for one to two weeks to remove the softer surface on top of the CGF stem and impurities, and then placed in sunlight for three days to remove the moisture. In this paper, the main emphasis is on comparing raw fiber to treated fiber, so chemical treatment with 5% NaOH for 45 minutes, undisturbed, is carried out to ensure that the chemical interacts properly with the fiber to achieve the best results practicably. The single fiber tensile test showed that the tensile strength and young's modulus of alkali-treated CGF significantly improved, while thermal stability and degradation temperature decreased hemicelluloses and increased cellulose, demonstrating that chemical treatment improves mechanical properties. This study concludes that alkaline-treated fiber would outperform untreated fiber. Surface roughness is dramatically modified, according to the SEM image. As a result, fiber can

be used to make car doors, interiors, dashboards, headliners, spare tire covers, spare wheel pans, seatbacks, and other parts. This is a good option for preparing a helmet and car seating.

Satyanarayan Panigrahi, et.al., (2007) Chemical treatment is discussed in detail in this paper, and various chemical types and methods are used to conduct the treatment to improve the mechanical, chemical, physical, and morphological properties of the fiber. It may increase the interface adhesion between the fiber and the matrix, as well as decrease fiber water absorption, as described below. As a result, chemical treatments can be used to alter the properties of natural fibers. Peroxide, silane, acetic acid, acrylic acid, sodium hydroxide, melted coupling agents, isocyanates, potassium permanganate, and other compounds are known to facilitate adhesion by chemically coupling the adhesive to the content. The efficient adhesion properties of fiber modification methods described in this paper vary from the raw fiber.

Suyambulingam Indran, et.al., (2018) This paper outlined the single fiber tensile test methodology, which included testing with various gauge lengths of fiber ranging from 10mm to 50mm and testing by the UTM for every single fiber of 30 samples. The physical measurements of the fiber are taken with different diameters, and the diameter is measured at various points to obtain the average diameter of the fiber, which is then used to carry out the tensile test and determine the tensile value. Optimum composite strength was achieved with 30 weight percent fiber content for a fiber length of 40 mm, which is higher than most other natural fiber-reinforced polymer composite materials. SEM photographs of the broken samples are used to examine failure at high fiber material. The higher fiber content caused further failures due to fiber pull out and debonding due to an inappropriate matrix and fiber-to-fiber contact due to a small gap between the fibers, as shown by the article.

Asma El Oudiani BenSghaier, et.al., (2012) The chemical treatment with different alkali on the same fiber is performed in this journal. After the chemically treated fiber has been compared, these fibers are treated with NaOCl and NaOH at different

percentages and used between 1 and 30% NaOH and 5 to 20% NaOCl. using NaOCl for alkali treatment, fiber properties might change or show no difference. The alkaline treatment with NaOH, on the other hand, had a huge impact.

Muhammad Ramzan Abdul Karim, et.al., (2020) This paper has demonstrated the effect of sodium carbonate on natural fibers using short bamboo fibers. The alkaline treatment takes about 6 hours at room temperature and uses 5% sodium carbonate. In this paper, we'll go over how to conduct a chemical treatment and how to select the right chemical for the job. The results of using sodium carbonate showed that the treated fiber had a 15% higher tensile strength than the raw or untreated fiber. The FTIR test showed that impurities such as amorphous compounds were reduced and the cellulose content was increased, which improved the fiber's tensile properties. When TGA findings were analyzed, it was discovered that the treated fiber degraded 15% faster than the untreated fiber. The interlocking and bonding of the fiber with the composite matrix are strengthened as a result of the chemical effects and the composite material gains strength as a result.

Brígida.A.I. S, et.al., (2010) Tests used in this study are TGA, Xrd, FTIR, SEM, and other analyses used to examine the properties of fibers. SEM images verified that the surface chemical composition is unaltered, but the wax and fatty acid are effectively extracted when treated with hydrogen peroxide. The loss of hemicelluloses in the treated fiber with NaOCl/NaOH was confirmed by FTIR and chemical composition tests, while this fiber showed a greater improvement in cellulose content and decreased thermal stability. The morphology of treated fiber surfaces with NaOCl is close to that of raw natural fiber surfaces.

Na Lu, et.al., (2013) Effect of two different chemical modifications is discussed by this author. This research looks at the effects of NaOH and silane treatments on hemp fiber for thermal and mechanical properties of higher density hemp composites. The findings showed that treated fiber has higher thermal stability than untreated fiber

and that as fiber loading increased, thermal properties and composite stability decreased. Dynamic analysis reveals that treated composites had higher mechanical properties in comparison to untreated composites. Silane-treated hemp composites had a higher storage modulus than NaOH-treated hemp composites, indicating a stronger fiber-matrix interface in the paper.

Harwood.R, et.al., (2009) Introduction to Testing of natural fibers for quality and fitness of fiber with considering the length, diameter, strength, colour, elongation, and impurity contents in the fiber which is related to agro-textiles. Fiber properties are divided into two categories in this article. The first group is concerned with fiber quality, such as length and fitness, while the second group is concerned with non-fiber pollutants created or retained in fiber during the extrusion process, such as dirt, grease, and vegetable matter. As previously mentioned, the identification of containment that causes disruption must be understood to quantify fiber properties perfectly. Testing is very important in development since various methods are developed for quickly and reliably obtaining test results, and the costs involved are often less important than the quality of the data produced. Many approaches have been taken from industrial standards to execute standard procedures agreed by ISO and various international organizations for various types of standardized procedures to follow, as stated. Various types of experiments were carried out in this article, and the results were analyzed.

Rowell, et.al., (2008) The journal provides information on natural fiber, ranging from the fundamentals to current study. A comprehensive flow chart of natural fiber has been published. Going through this paper exposed to fiber types and what kind of fiber we have taken for our study, bast fiber is identified along with all kinds of fiber extraction from all means, such as seeds, grass, stem, branch, core fiber from the middle of the stem, so everything is experimented with, and a lot of research work is done to differentiate properties between sources observed during authors' journey has just scratched the surface of the natural fibers universe. In the future, a new kind of natural fiber would need to be considered for research to conduct applications that are only

feasible with certain fibers. From the research point of view, some references have been given.

Indran.S, et.al., (2015) This study looks into the classification of *Cissus quadrangularis*. With its excellent properties, stem fiber offers a new way for fiber to compete with synthetic man-made fiber. The density is lower than that of common synthetic fibers, etc., and the basic results are 20% better. Tensile strength and Young's modulus are substantially higher than any other natural fiber ever studied, and they are almost comparable to commonly used synthetic fibers. When chemical treatment is performed, the bonding strength is improved. Increased fiber roughness assists in better composite manufacturing. Its stability is verified by thermal analysis. With some advances, this CQSF will substitute and equal the properties of synthetic fiber. Thermal stability of CQSF or any natural fiber is detail between 90°C to 100°C removal of wax and moisture content observed degradation of hemi-Cellulose content, lignin, and glycosidic links of cellulose start at 200°C and fiber starts to burn at 340°C maximum mass losses is observed in this stage. Next and gradual drop from 400°C to 710°C is observed as degradation of oxidative elements and remaining lignin content is removed, at remaining mass are calculated at end.

Saravanakumar.S. S, et.al., (2014). In this journal the bark of *prosopis juliflora* plant was chemical treated and test like XRD, TGA, chemical composition test have been performed and studied in detail that at optimal percent of chemical treatment conditions influence the degree of swelling that effects the lattice transformation of the cellulose present in fiber with virtual improvement of 17.23wt% and decline in hemi cellulose and lignin by 12.12 wt.% and 5.02 wt.% respectively .The low wax and moisture content is observed ,interesting result is that ash content increased by 8.20wt% which is due to removal of amorphous composition elements when fiber is treated with optimal conditions and change in fiber structure is concluded by FTIR ,XRD and TGA.

Di Liu, et.al., (2009). The *nelumbo nucifera* fiber with isolated walls and leaf stale of unique shape have been studied to understand the morphology and cellulose structure of the fiber by this journal. FTIR is performed to identify the structural information or change of structure, it is conducted on four samples at four stages with certain variations like fiber of Nn and tracheary element from leaf are taken. The results shown that there is no large change in FTIR spectra for these samples, excluding the absorption bands with change in relative intensity. The peak at 3361 cm^{-1} and 2900 cm^{-1} was ascribed as O-H or N-H stretching vibrations and CH and CH_2 stretching vibrations respectively. The various peaks at 1734 cm^{-1} , 1033 cm^{-1} and 1640 cm^{-1} are observed and attributed to different stretching of methyl ester, carboxylic acid, acetyl and oxygenous groups belong to pectin, hemicellulose and cellulose and lignin chemical composition of fiber. The longitudinal shape, cross sectional and microstructure images of fiber were observed using SEM analysis. The *Nu* fiber displayed certain properties at different growth period, at primary or first stage there is no link fiber and not arranged. the fiber is arranged at isolated manner in second stage, at third stage linkage appears with small quantity irregularly., the quantity of the links increases between fiber and occurrence of wax is observed from this longitudinal view. some large voids and cluster are presented on the brittle surface of Nn with fractures, fiber with different fibril sizes were displayed in cross sectional and micro structure images of fiber from SEM analysis are observed.

Suryanto, et.al., (2014) this journal was study of morphological, structural and chemical properties of treated and untreated *mendong* fiber to find the chemical or functional groups of fiber FTIR is performed, for structural and morphological study SEM and XRD are performed. The XRD spectrum contains three peaks at $2\theta = 16.31^\circ$ and 22.35° the presence of amorphous content like lignin and hemicellulose content is observed due to broad peak at $2\theta = 16.31^\circ$ The intense peak at $2\theta = 22.35^\circ$ was due to presence of cellulose and closer compositions to cellulose are observed. After chemical treatment by 5% of sodium hydroxide for 2 hours no change in crystalline structure and index showed by diffraction (2θ), but increase in intensity is observed due to increase of crystalline cellulose and removal of amorphous parts is observed.

CHAPTER-3

AIM AND SCOPE OF THE PROJECT

3.1 AIM OF THE PROJECT

This project is more emphasized on replacing synthetic composites with renewable and biodegradable materials which suits our ecosystem, the aim is to prove a natural fiber compatible to replace them is discussed in detail below.

- To investigate up to what loads the fiber can withstand for treated and untreated fiber.
- To understand whether using this in automotive applications is feasible.
- To obtain the characteristic data of *Coccinia Grandis* and its respective applications.
- To study the structure using the micrographs from Field Emission Scanning Electron Microscope (FESEM) this visualizes very small topographic details of Fiber.
- To perform EDAX (Energy Dispersive X-ray Analysis) so that elemental composition of the of alkali treated *Coccinia Grandis* can be represented.
- To perform XRD (X-Ray diffraction) analysis so that information of the crystalline structure can be compared.
- To perform Thermo gravimetric analysis (TGA) an analytical technique obtaining the information of heat resistance and degradation of samples heated at a constant rate by monitoring the change in mass accordingly.

- Chemical bonding information of the fiber is known for us in Fourier Transform Infrared Spectroscopy (FTIR) analysis.

3.2 SCOPE OF THE PROJECT

- The outcome will be a detailed report of all the characteristic properties of *Coccinia Grandis* fiber with comparative results from both untreated and alkali treated fiber for single fiber tensile test, XRD investigation, TGA analysis, the micrographs from FESEM, chemical bonding of the fiber is taken from FTIR analysis and EDAX.
- The data obtained through above analyses will be useful to all the automobile, aero and industries which deal with natural fiber composite.
- The scope is to replace traditional synthetic materials which are non-biodegradable to natural materials which are biodegradable in the world.
- The outcome will be the advantages of raw and alkali treated fiber composites in the field of automobile applications.
- The above given analyses will fetch a huge amount of information in knowing the *Coccinia Grandis* applications better.
- The chemical analysis gives us the impact of the alkali for a particular amount of time that the fiber was treated.
- A comparison between XRD and FTIR graphs of raw and alkali treated fiber is done and a better fiber is recommended.

CHAPTER – 4

MATERIALS AND METHODS

4.1 METHODOLOGY

The detailed process of our project is explained below Fig 4.1.

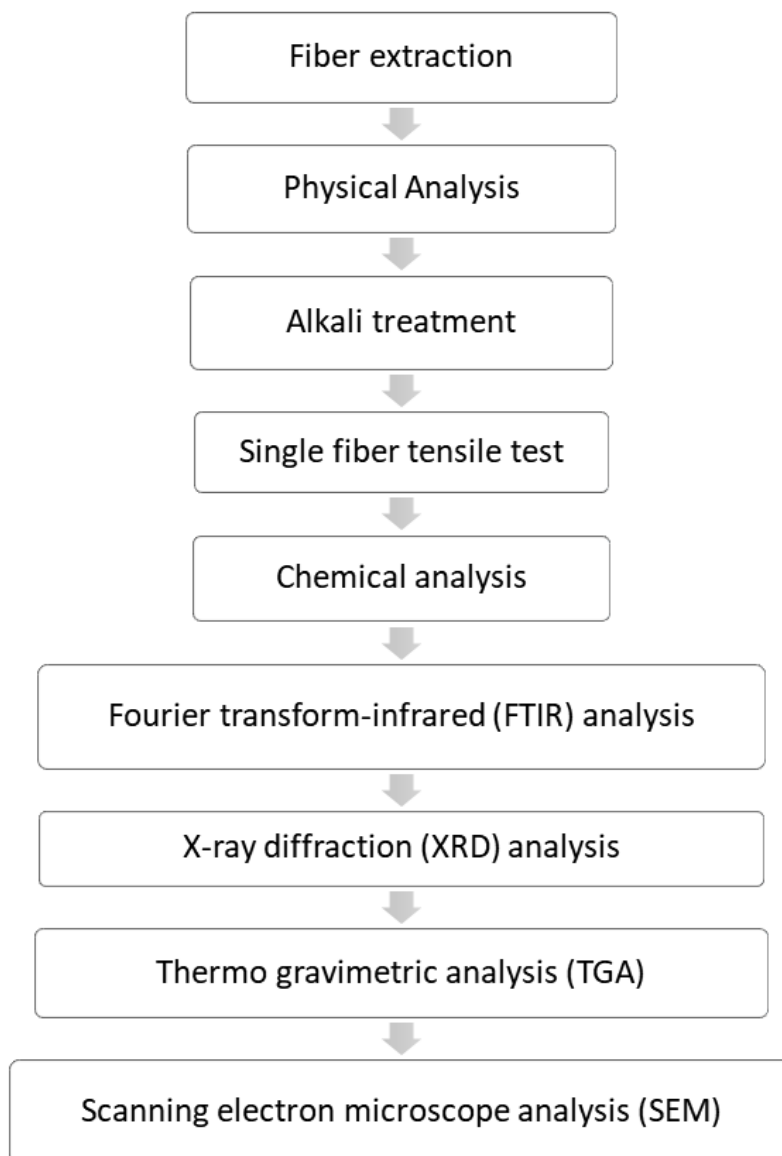


Fig:4.1 : Workflow flow chart

4.2 MATERIALS REQUIRED

Coccinia Grandis fiber, the powdered sodium carbonate (Na_2CO_3), digital micro meter, three beakers, and distilled water.

4.3 FIBER EXTRACTION

The *Coccinia Grandis* fiber stems were collected from the village karai, located in Tamilnadu, India. The stem extract is properly cleaned and washed to be free from dirt or soil containment, the water retting process was used to extract the fiber from the stem. The stems were kept underwater undisturbed in a container for two weeks to remove the outer hard skin or surface from the stalk through the retting process. After primary walls or skill was peeled and separated so that the inner fiber layer is acquired. The fiber with impurities is collected, rinsed, and washed in clean water until the fiber is free of the skin or outer walls of the stem, wet fiber is collected and placed in direct sunlight for two to three days to remove moisture content from the fiber. the extraction process of *Coccinia Grandis* plants and fibers is portraited in fig 4.2.



Fig:4.2 : Extraction Images;

(a)CGF plant;(b) Stem of CGF;(c) Stem immersed in water;(d) Intermediate state of retting process;(e) Wet extracted fiber;(f) Dry fiber.

4.4 PHYSICAL ANALYSIS

The natural fiber diameter is very complicated to measure since the fiber is not uniform in shape. However, profile shapes and diameters of the raw and alkali treated CGF were resolved by using a digital micrometer with the ability to measure up to 0.001mm. Thirty samples of each gauge length were measured in both raw and alkali-treated CGF, as shown in fig 4.3. Measuring fiber diameter concluded that fiber was almost circular in cross-section. The measurements were taken in three different random places (top, middle, and bottom) to estimate the fiber's mean diameter.



Fig:4.3 : Digital micro meter

4.5 DENSITY MEASUREMENT

The density of *coccinia grandis* is measured by the concept of Archimedes principle by using immersion liquid methanol as measuring fluid.

$$\rho_{CGF} = \frac{m_2 - m_1}{[(m_3 - m_1) - (m_4 - m_2)]} \times \rho_m$$

..... equation 1

where ρ_{CGF} is the density of the CGF (g/cm³), ρ_m is the density of the methanol (0.791 g/cm³), m_1 is the weight of sample container in air, m_2 is the weight of sample container in the methanol, m_3 is the weight of sample container plus chopped fibers in the air, and m_4 is the mass of the sample container plus chopped fibers in methanol.

4.6 SINGLE FIBER TENSILE TEST

The tensile properties of the individual fibers were tested under ASTM D3822-07 international standards using Instron 5500R fig 4.5 universal testing machine. Raw CGF with gauge length ranging from 10mm to 50mm with increments of 10mm, 30 samples fig 4.4 for each gauge length are tested at a 5mm/min crossing rate with the load of 1N to estimate the average tensile properties of an individual fiber.

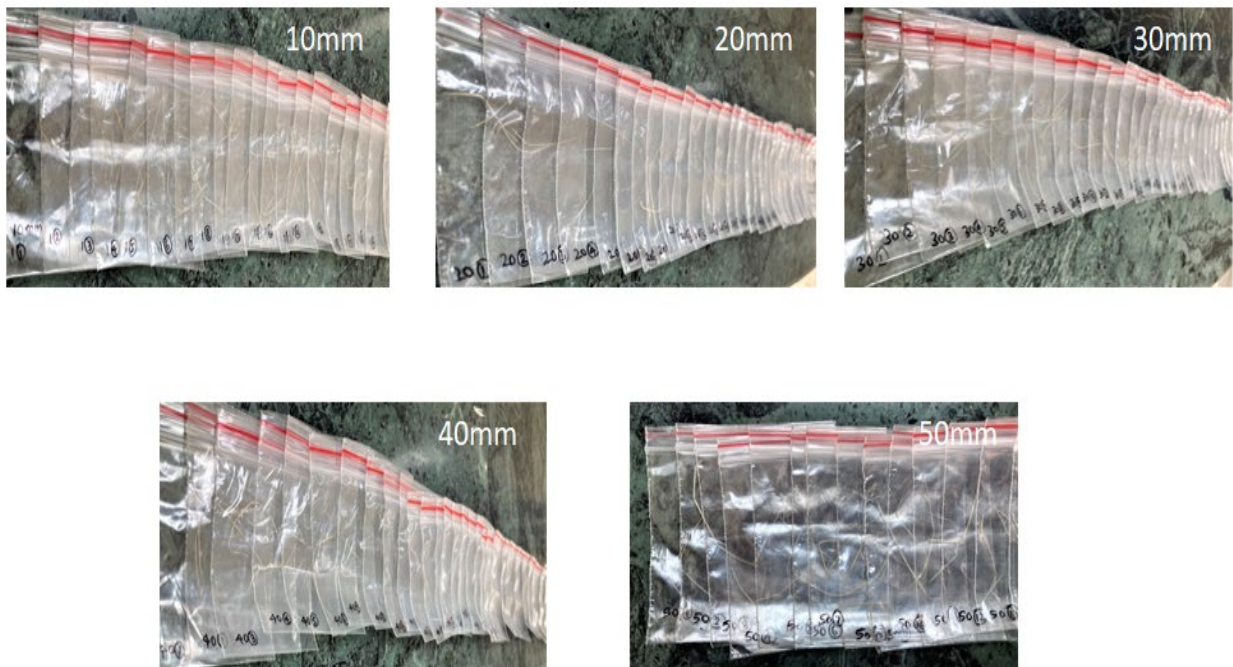


Fig 4.4;. samples for single fiber tensile test

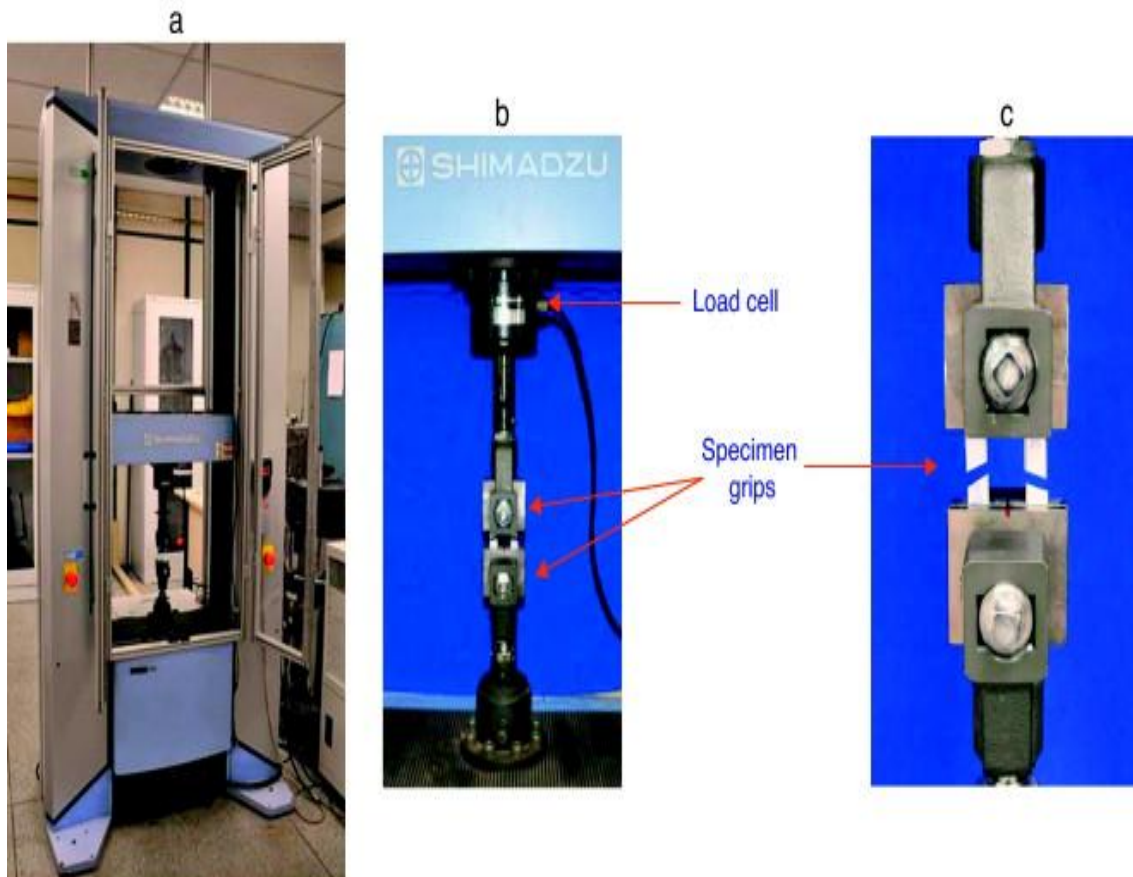


Fig:4.5 : Instron 5500R Machine

4.7 ALKALI TREATMENT

After a fiber tensile test of untreated fiber for better improvement in tensile strength of the 10mm gauge length, we chemically treated the dried CGF with sodium carbonate (Na_2CO_3) for 6 hours as optimum time at room temperature for the chemical to react with the fiber. The chemical and distilled water are mixed weight to volume ratio of 1:20. This treatment is done with three different percentages of chemical (5%, 10%, and 15%) for 30 samples of each concentration, and the fiber is heat-treated in the oven for 100 °C for two hours and dried in sunlight for one day to remove the moisture content of the fiber. The whole process of chemical treatment is shown in figure 4.6. Every sample is packed separately in sealed covers and sent to a single fiber tensile test. Based on the chemical-treated tensile test results, 22 grams of fiber is treated with 15% of sodium carbonate and sealed in polyethylene bags for further tests.

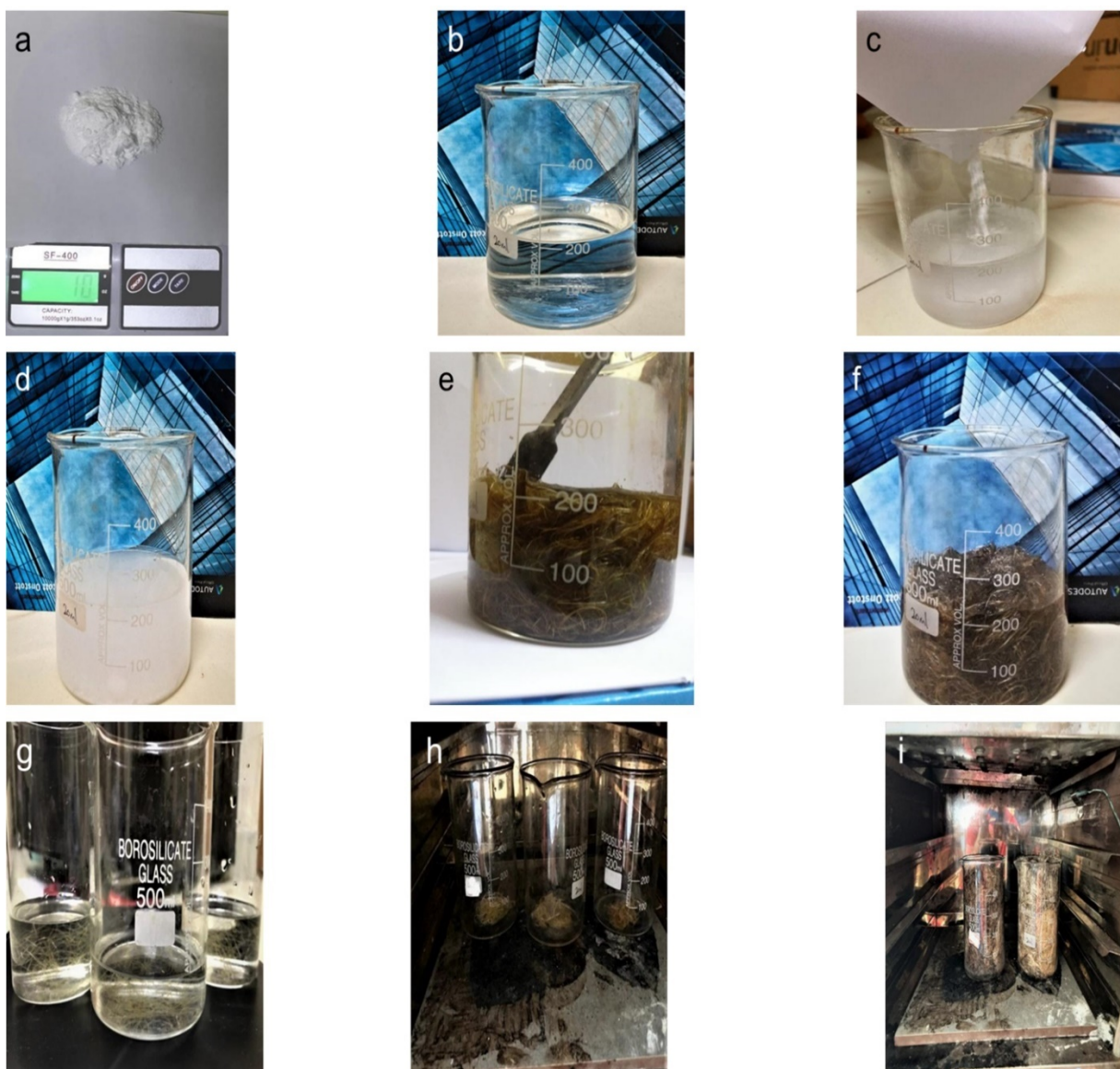


Fig:4.6 : Alkali treatment process;

(a) Chemical weighing; (b) 200ml distilled water; (c) Chemical mixture; (d) Dissolved chemical; (e) Fiber immersed in solution; (f) Fiber immersed for 6hours; (g) Single fiber chemical treatment with different concentrations; (h) Single fiber heat treatment; (f) Chemical analysis heat treatment

4.8 CHEMICAL ANALYSIS

To determine the composition percentages of wax, cellulose, ash, hemicellulose, lignin, moisture, and pectin contents in fiber the chemical analysis is performed on the raw and alkali treated *Coccinia Grandis* fiber by using suitable techniques. The Kurschner and Hoffer procedure is followed to calculate the cellulose content. The Goering and Van Soest method of the neutral detergent is used to determining the hemicellulose content. The Conrad method determines the wax content. For the ash content and lignin content, TAPPI's calculation standards procedure was used [2]. Ten grams of raw and treated CGF were placed in the air oven to eliminate moisture at 100°C for two hours. After samples are taken from the oven and their weights were measured, determines moisture content of raw and alkali treated fiber by using formulae mentioned below.



Fig:4.7 : Fiber for chemical analysis

$$\text{Moisture content} = \frac{[(\text{Sample weight before drying}) - (\text{Sample weight after drying})]}{(\text{Sample weight before drying})}$$

.....equation(2)

4.9 FTIR ANALYSIS

FTIR is performed in (Fig 4.8) to identify the structural information or change of structure and to identify the chemical functional groups linked to various cellulosic compounds. both raw and treated natural fibers tested using infrared spectra to observe the peaks with the particular intensity that attributes to different stretching of compounds. The preparation of the sample is made by using mortar and pestle, the fiber is grounded into smooth powder and mixed with KBr powder in a 1:10 ratio to form a thin film or pellet for analysis. The graph was plotted between the percentage of transmitted infrared spectra intensity and the wave number from 4000 cm^{-1} to 500 cm^{-1} . The resolution of 2 cm^{-1} with 25 number of scans by using the Happ-Genzel function the FTIR analysis is carried out.

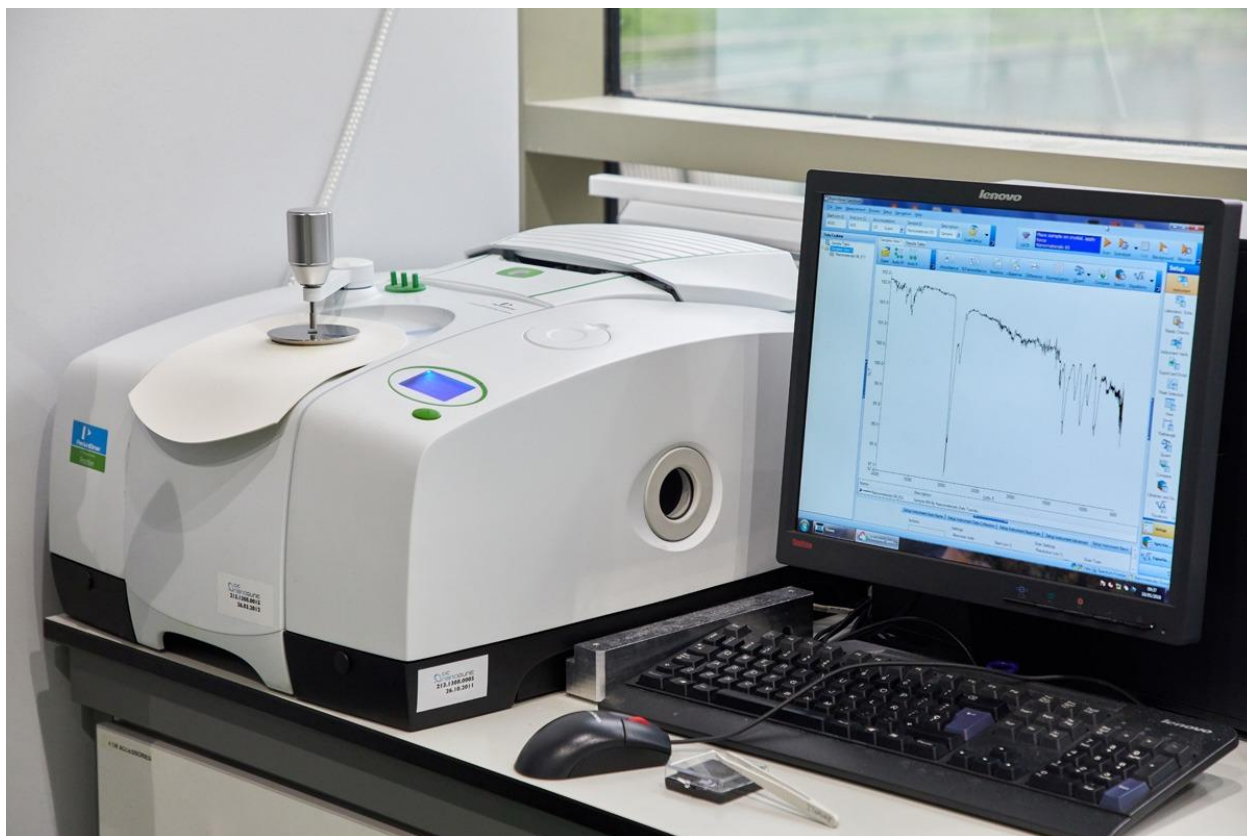


Fig:4.8 : FTIR operation machine

4.10 XRD ANALYSIS

XRD is the appropriate technique to determine the impact of alkali treatment on natural fiber and used for determining crystalline index and crystallite size of raw and surface modified natural fibers. The investigations were carried out by the X'PERT-PRO diffractometer Fig(4.9) with the automatic data storing capability. An X-ray detector rotated to scan between 10° to 80° (2θ angle) at a speed of 1° per min. The generator was functioning at 40 kV and 30 mA at room temperature of 25°C and segal equation is used to calculate the Crystalline index (CI) and crystallinity (%Cr) respectively,



Fig:4.9 : XRD operated image

4.11 TGA ANALYSIS

It is essential to check the thermal stability of the natural fiber to ensure the fitness for high temperature application usability. The sample is prepared 10mg of raw and treated fiber powder. Thermogravimetric analyses of untreated and treated CGF

were performed under nitrogen (N_2) environment using crucible Al_2O_3 in TGA Q50 Model shown in Fig(4.10). Initially masses of the samples were precisely measured at room temperature then temperatures of samples were increased from room $0^\circ C$ to $1000^\circ C$ at a constant heating rate of $10^\circ C$ per minute simultaneously. Loss of Mass were noted for every $1^\circ C$ rise in temperature then graphs (TG) were plotted between temperature ($^\circ C$) and weight percentages



Fig:4.10 : TGA equipment

4.12 SEM ANALYSIS

The SEM analysis of a natural fiber is essential to predict the surface roughness. Necessary microscopic images of the treated and untreated CGF were obtained using supra-55 CARL Germany, Scanning Electron Microscope (Fig 4.11). Heated tungsten filament was used as an electron source with magnification of 12-90,0000X, working distance <10mm and nominal resolution of 1.5nm at 10KV and WD=2mm. the beam of electrons focused on the sample's surface to produce the topographic image of the fiber with different orientation like cross sectional, longitudinal and micro structures of fibril with various sizes.

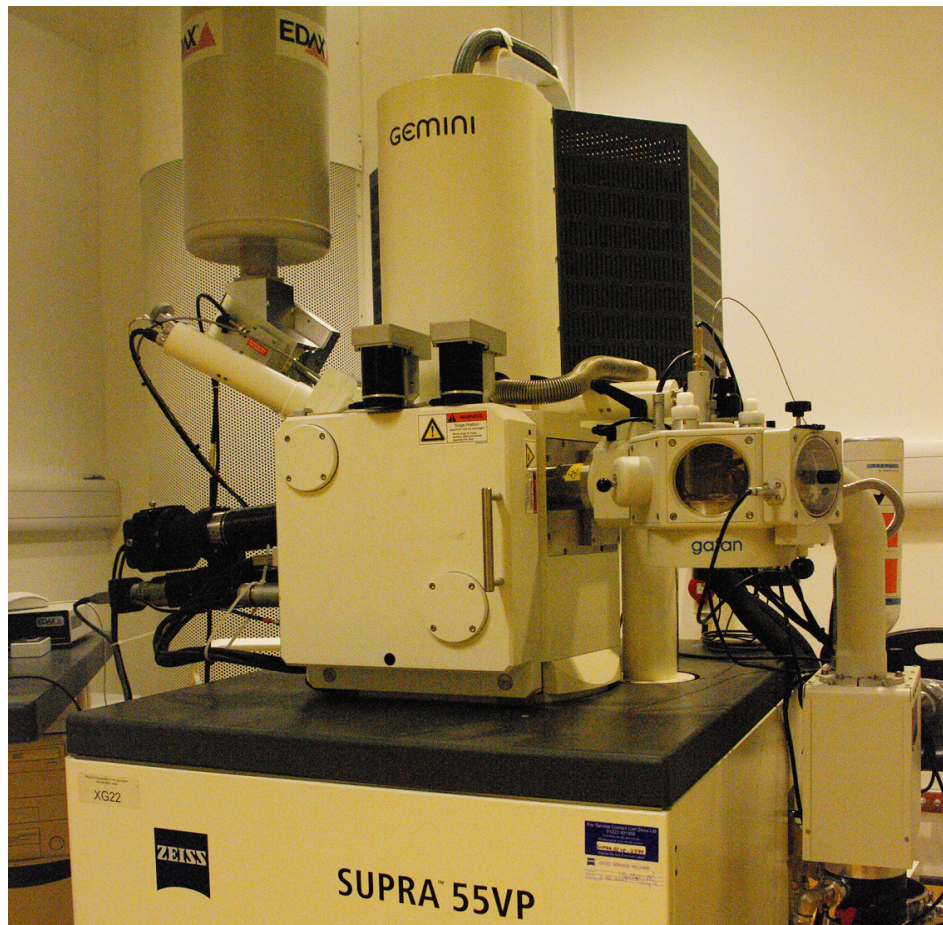


Fig:4.11 : SEM machine

CHAPTER-5

RESULTS AND DISCUSSION

5.1 CHEMICAL ANALYSIS

The mechanical, resistant, and thermal properties of the fiber is an influence of the chemical composition. The comparison between the treated and untreated fiber chemical compositions is summarized in table 5.1, The removal of wax and moisture content is observed in sodium carbonate treated fiber with increment in cellulose content is observed [29]. The Ash content increment illustrates the removal of the amorphous substance in chemical treated CGF, optimal percent of chemical treatment conditions influence affects the lattice transformation of the cellulose present in fiber with the improvement of 70.96% to 73.42%, high cellulose content will ensure the better tensile properties [4] and decline in hemicellulose and lignin from 28.74% to 17.68% and 12.19% to 11.95% respectively, the hydrolysis of hemicellulose and some links of cellulose in glycosidic bonds resulted in the parting of lignin-hemicelluloses bond which increased solubility of hemicellulose in water and lignin in alkali solvent forming a solid residue of cellulose due to chemical treatment is observed [5]. The low wax and moisture content are observed, ash content increased by 25% gain which is due to the removal of amorphous compositions when the fiber is treated at optimal conditions [30].

Table:5.1 : Chemical analysis results comparison

CHEMICAL COMPOSITION CONTENT	UNTREATED . wt. %	TREATED . wt. %
Cellulose	70.96	73.42
Lignin	12.19	11.95
Wax	0.52	0.44
Ash	2.19	2.74
Moisture	8.48	5.15
pectin	5.38	6.38
Hemi cellulose	28.74	17.68

5.2 TENSILE STRENGTH

5.2.1 *Untreated Fiber*

The untreated CGF tensile test results showed that the 10mm gauge length had the highest tensile strength of 304.17 ± 40.25 MPa. The average tensile strengths are graphed in Fig 5.1; as well as a quantitative comparison of Mechanical properties in table 5.2. The irregularity in mean diameter will affect the sectional area of the fiber that influences the tensile strength and young's modulus. The modulus of CGF raises with increased gauge length as expected for the all cellulosic fiber, the strain to failure at F_{max} decreases with increasing gauge length due to the distributed flaws of fiber as its length and volume increases [28].

Table:5.2 : Mechanical properties of Untreated CGF

S.NO	GAUGE LENGTH mm	MEAN DIAMETER mm	TENSILE STRENGTH MPa	STRAIN	YOUNG'S MODULUS GPa
1	10	0.0739 ± 0.0028	304.17 ± 40.25	0.0457	7.945±0.68
2	20	0.0776 ± 0.0018	206.69 ± 20.56	0.0238	9.306±1.27
3	30	0.0819 ± 0.0035	218.04 ± 25.65	0.0206	11.205±2.03
4	40	0.0803 ± 0.0029	182.94 ± 18.53	0.019	11.233±1.95
5	50	0.0791 ± 0.0041	223.16± 26.40	0.0171	14.702±2.42

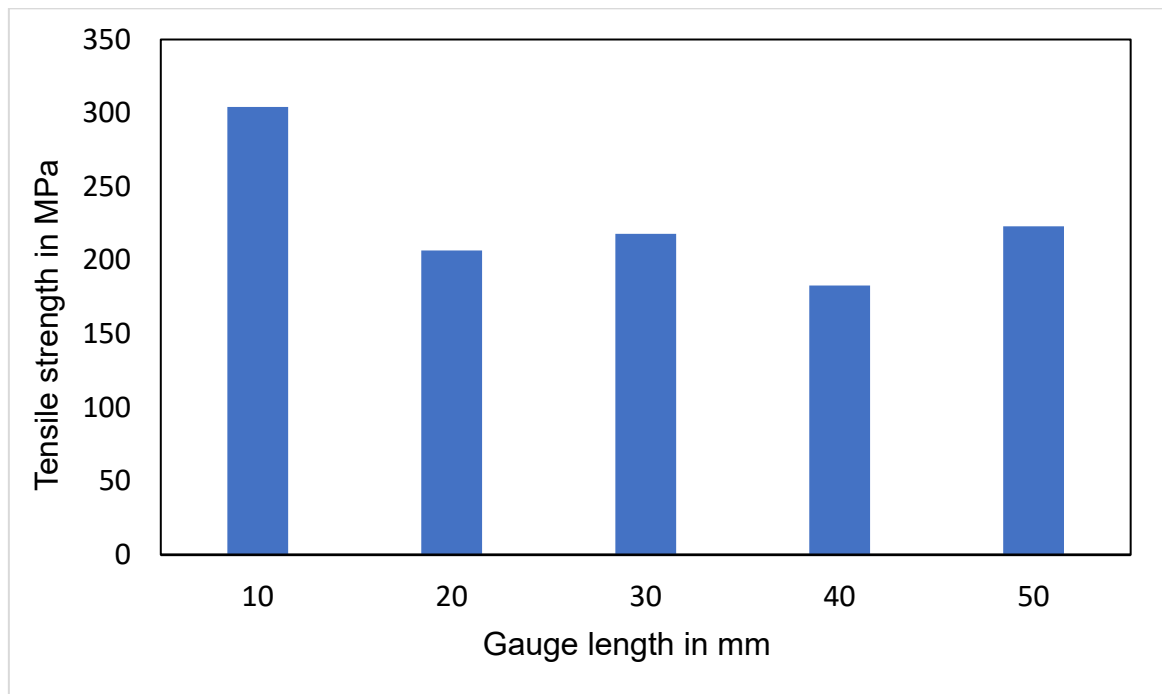


Fig:5.1 : Untreated CGF Tensile Strength values Comparison

The tensile properties of CGF are also dependent on its age, plant source, the procedure used for the fiber extraction, and microfiber structure as all-natural fibers, when the crack initiated from the bigger void or flaw lead to fiber breakage and failure. As the Gauge length increases, the probability of flaws gradually raises, and those both

simultaneous causes sudden failure at higher gauge lengths [12]. There is the reason for the 10mm gauge length to have a higher tensile strength of 304.169MPa compared to other gauge lengths of 20mm, up to 50mm. The elongation of break at F_{max} of raw and alkali-treated CGF was between 1.7% to 4.57% and 3.32% to 4.717%. The elongation of break of natural fibers is direct proportional to microfibril angle and tensile strength and young's modulus is inversely proportional to microfibril angle. So, fiber with least microfibril angle is the favourable property for composite preparation [10]. When a load is applied to the fiber, shear stress distributed along the fibre length and ends of the fiber shows us change in length and breaks the fibre after its elastic point. The tensile properties of the fiber depended on influencing factors such as fibre orientation, length and loading [19].

5.2.2 Treated Fiber

The tensile strength of the chemically treated CGF was compared, and it has been determined that 15% sodium carbonate treated fiber of 10mm length had the highest tensile value of 341.295 MPa. The tensile value, on the other hand, fell from 5% to 10%. Graphical interpretation of the results in Fig 5.2 and values was tableted in table 5.3.

The elongation, strain, and tensile properties of the fiber increased as the chemical composition increased, while the mean diameter decreased. As the chemical percentage increases, the fiber reacts with the chemical, which eliminates impurities and hemicellulose material. This demonstrates that adding sodium carbonate to CGF enhances their properties [21]. As the result alkali treated fiber exhibiting greater tensile strength than untreated fiber.

Table:5.3 : Mechanical properties of Alkali treated CGF

S.No	GAUGE LENGTH mm	ALKALI %	MEAN DIAMETER mm	TENSILE STRENGTH MPa	STRAIN	YOUNG'S MODULUS GPa
1	10	5	0.0885±0.0024	258.18 ± 25.48	0.0333	8.472 ± 1.42
2	10	10	0.0957±0.0049	243.36 ±18.53	0.0444	6.495 ± 0.85
3	10	15	0.0795±0.0018	341.30 ± 39.52	0.0471	7.984 ± 1.07

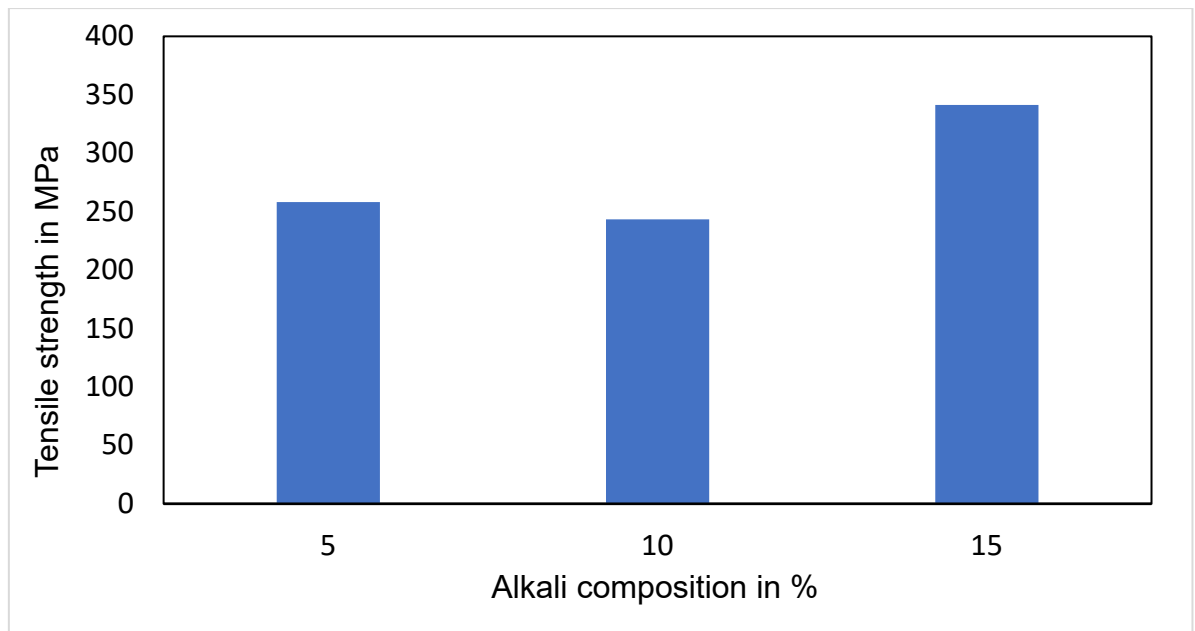


Fig:5.2 : Alkali treated CGF Tensile Strength values Comparison

5.2.3 Density Measurement

The density of the fiber increased from 0.90 g/cm³ to 1.06 g/cm³ by chemical treatment when compared with raw or untreated CGF fiber due to pores and voids are filled with micro molecules during reaction with sodium carbonate. the density of CGF is low compared to other natural fibers like CQSF, 1.22 g/cm³ [17]; kenaf ,1.50 g/cm³[22].

5.3 SEM ANALYSIS

To evaluate the effect of sodium carbonate on the surface of fibers. The SEM analysis was used to examine treated and untreated fibers. Fig 5.3 illustrates untreated fibers with tiny voids, helical fibrils, and cracks. This demonstrates the presence of impurities, lignin, wax, pectin, and oil on the surface of raw or untreated CGF, with oils and wax acting as a protective layer of the fiber. As the result of chemical treatment, oils, lignin, pectin, and impurities on the surface of the sodium carbonate treated fiber produced an improvement in roughness on the surface, which enhanced the bonding with the matrix or resins in composite preparation and improved the fiber texture, as seen in the micrograph of treated fiber in Fig 5.4. By observing the surface topography of treated fiber, the primary cell walls of the untreated fiber is dissolved during alkali treatment of 6h. Similar results were acquired by a other author using only Na_2CO_3 [21]. The structure changes of fiber has been analysed and observed by SEM images of treated and untreated CGF.

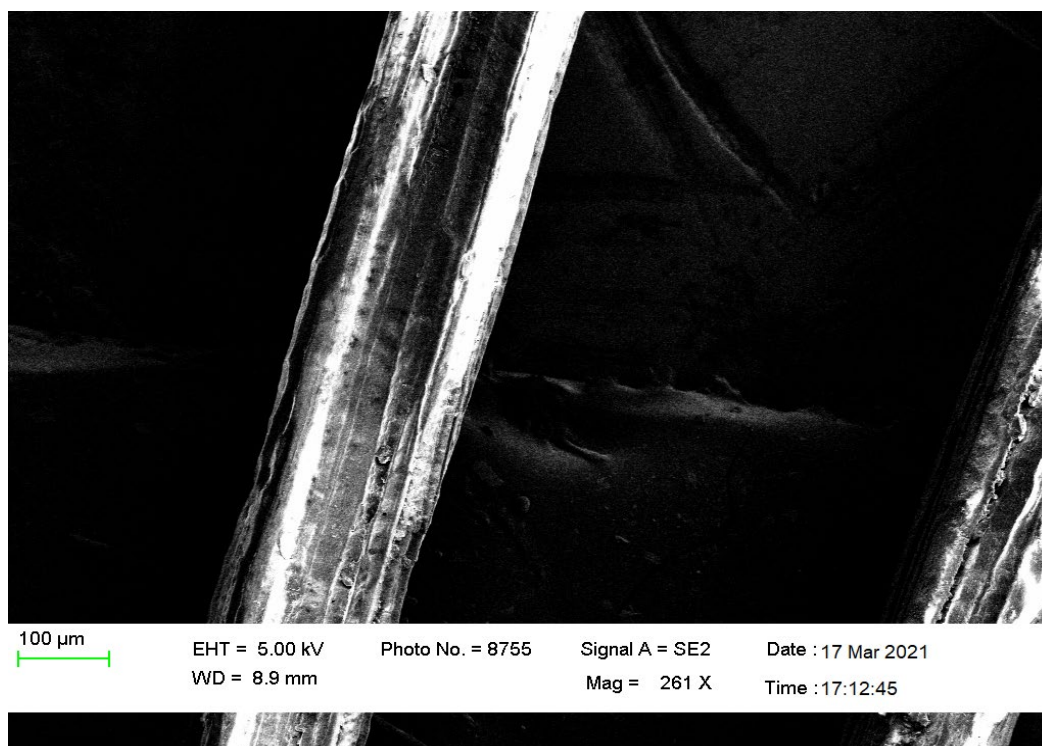


Fig:5.3 :SEM image of untreated CGF

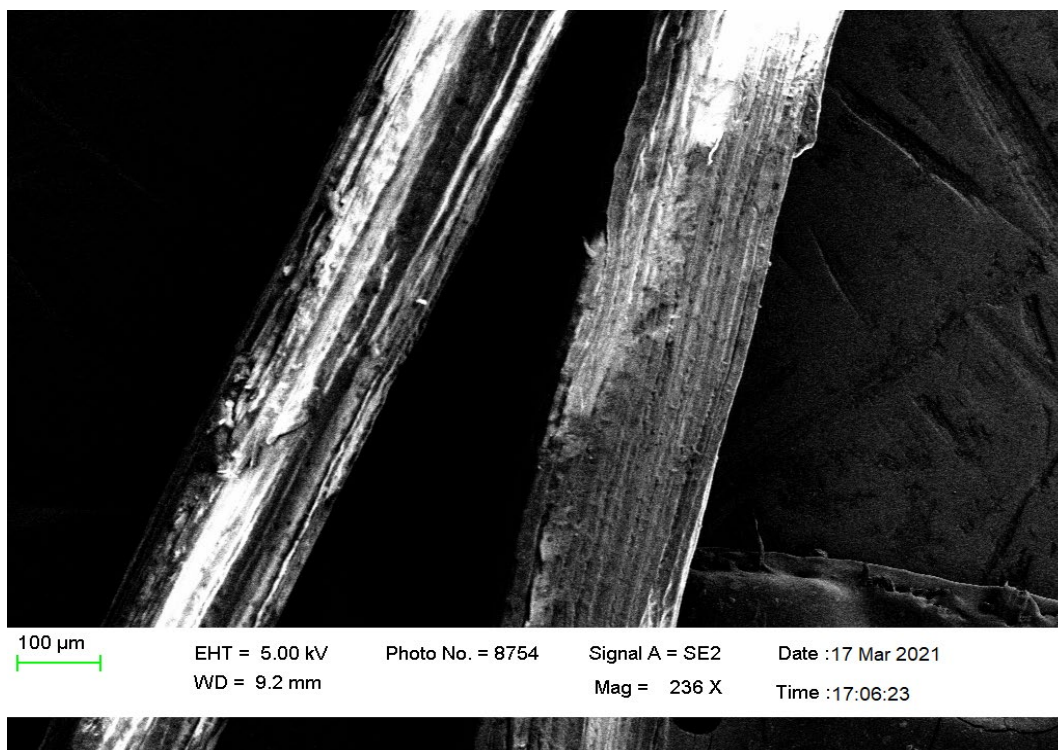


Fig:5.4 : SEM image of Alkali treated CGF

5.4 FTIR ANALYSIS

The FTIR test is used to determine the functional groups of the treated and untreated fibers, as well as their chemical composition. The FTIR analysis was completed, and the findings are depicted in Fig 5.5. Peaks can be found at 3289, 2905, 2320, 1728, 1641, 1501, 1427, 1246, 1139, and 1023 cm^{-1} in untreated fiber. OH, and CH stretching (cellulose) are responsible for the peaks at 3289 cm^{-1} and 2905 cm^{-1} , respectively [20].

A small peak at 2320 cm^{-1} is linked to wax material $\text{C}\equiv\text{C}$ stretching. The $\text{C}=\text{O}$ stretching content of hemicelluloses is represented by a noticeable peak at 1728 cm^{-1} [18]. The $\text{C}=\text{O}$ stretching of lignin content is responsible for the peak about 1641 cm^{-1} and 1501 cm^{-1} . The peak at 1427 cm^{-1} was due to cellulose stretching by CH_2 . $\text{C}=\text{O}$ stretching of hemicellulose material was connected to a peak at 1246 cm^{-1} . A visible peak at 1139 cm^{-1} is due to polysaccharide $\text{C}-\text{O}-\text{C}$ stretching [11].

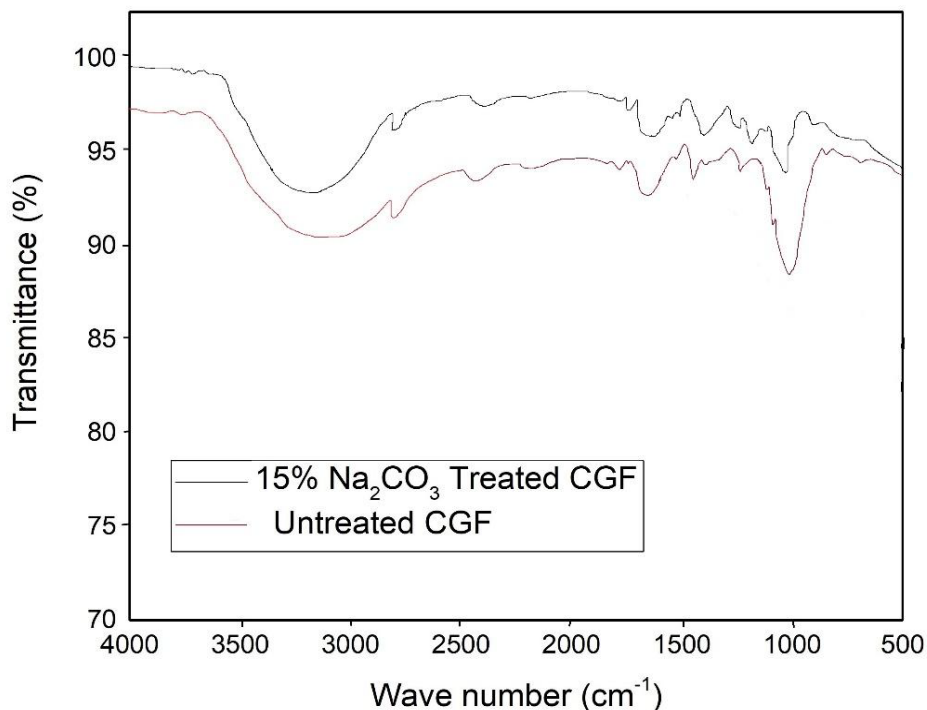


Fig:5.5 : FTIR analysis graph

The C-OH stretching of lignin content is responsible for the peak at 1023 cm⁻¹. The peaks at 1728, 1641, and 1246 cm⁻¹ were absent in alkali-treated CGFs, and the peak at 1023 cm⁻¹ was steadily reduced. It is to show that the removal of hemicelluloses and lignin from the chemically treated CGF confirms the chemical analysis findings.

5.5 XRD ANALYSIS

XRD analysis was used to assess the crystalline nature of *Coccinia grandis* and to determine the effect of the chemical treatment on the fiber cellulose crystalline, as shown in the Fig 5.6. After chemical preparation, the amorphous content of the fiber varies in CGFs. The efficacy of chemical treatment is calculated by the shift in crystalline index.

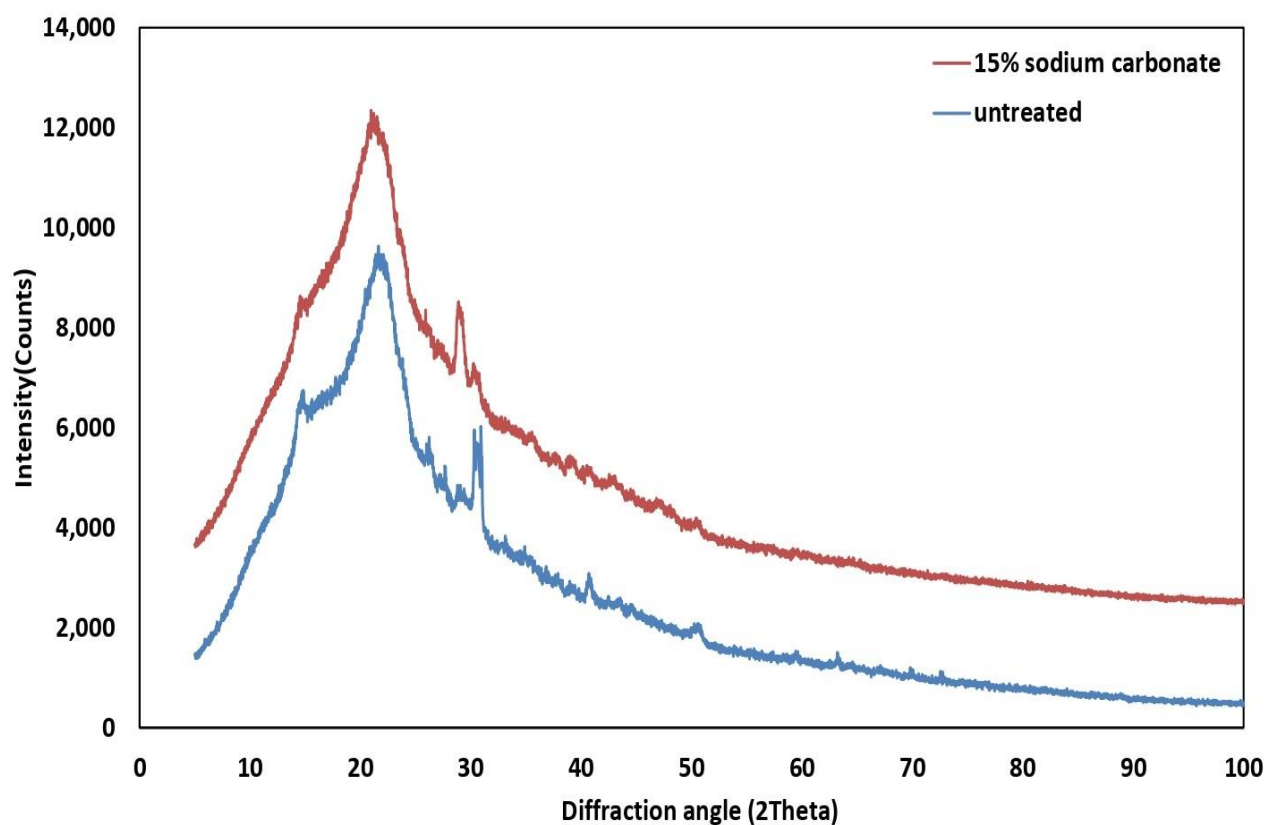


Fig:5.6 : XRD analysis graph

Fig 5.6 displays the XRD orientation of raw and chemically treated CGFs. It's easy to spot two major peaks about 14.8° and 21.6° of 20 degrees, which are common plant fiber peaks. There was a visible peak about 40.7° (0 4) that corresponded to the orientation and direction of the fibers [13]. The peak (21.6°) of sodium carbonate-treated CGFs was noticeably higher than that of untreated CGFs, indicating that the sodium carbonate solution penetrated the fiber and dissolved low molecular content such as hemicelluloses, wax, and lignin content from the fiber's surface, revealing the cellulose content of the fiber. The values of the crystalline index (CI) of raw and treated CGFs (52.17%, 57.64%) are calculated and compared [27].

5.6 TGA ANALYSIS

It is essential to check the thermal stability of the natural fiber to ensure the fitness for high temperature application usability. The sample is prepared 10mg of raw and treated fiber powder. Thermogravimetric analyses of untreated and treated CGF were performed under nitrogen (N_2) environment using crucible Al_2O_3 in TGA Q50 Model. The TGA and DTG curves of the untreated and treated CGF are shown Fig 5.7; 5.8. The first change in mass was noticed at 91°C ,5% mass losses which is occurred due to the removal of moisture content and wax content

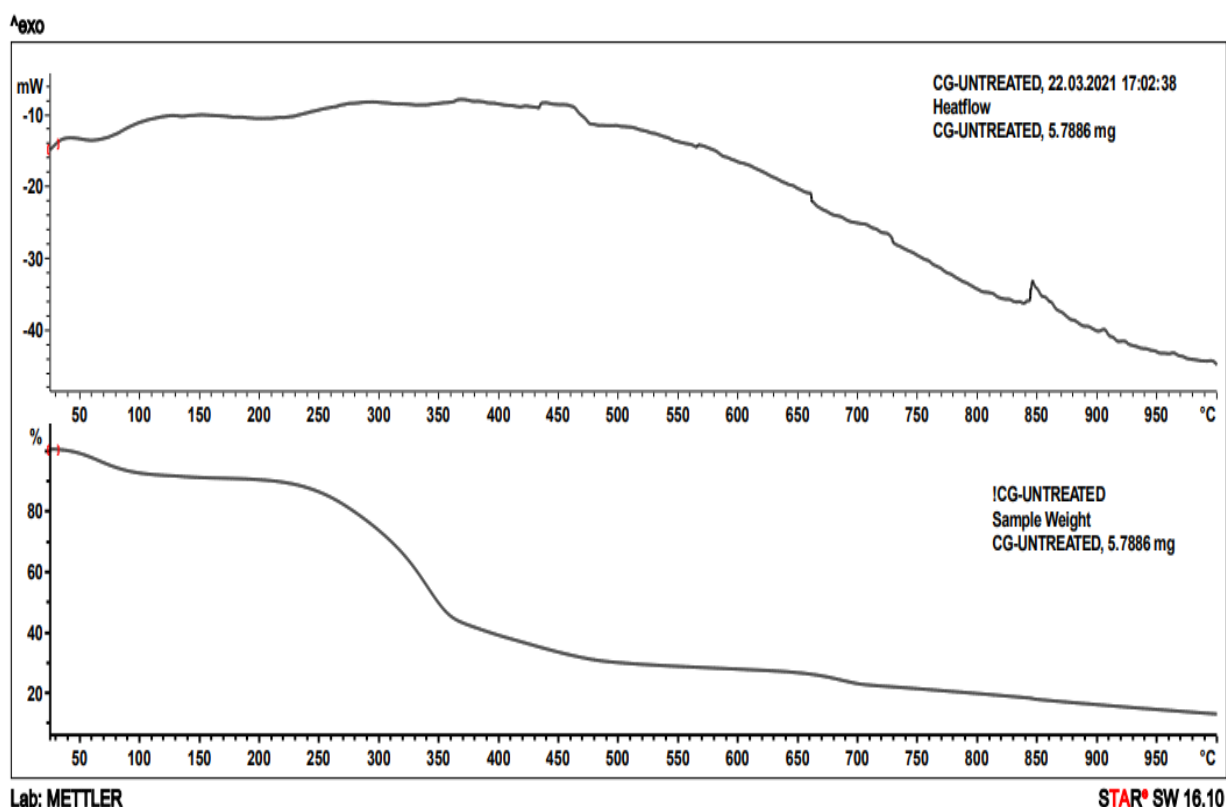


Fig:5.7 : Untreated CGF TGA

The second stage between 290 °C to 355 °C which is highest drop of 50% mass reduction indicates the removal or degraded hemicellulose content and glycosidic links of cellulose and that starts to burn the fiber which proves degradation of cellulose

content. Similar kind of drops and changes were reported for the other fibers like hemp, bamboo, kenaf and jute at 308 °C ,321 °C, 307 °C and 298 °C respectively [30]. The less noticeable gradual drops from 460 °C to 710 °C are the results of oxidative and lignin content degradation of the fiber. The 18.4% of mass was founded at the end of process at 999°C [17] is observed from results of TGA.

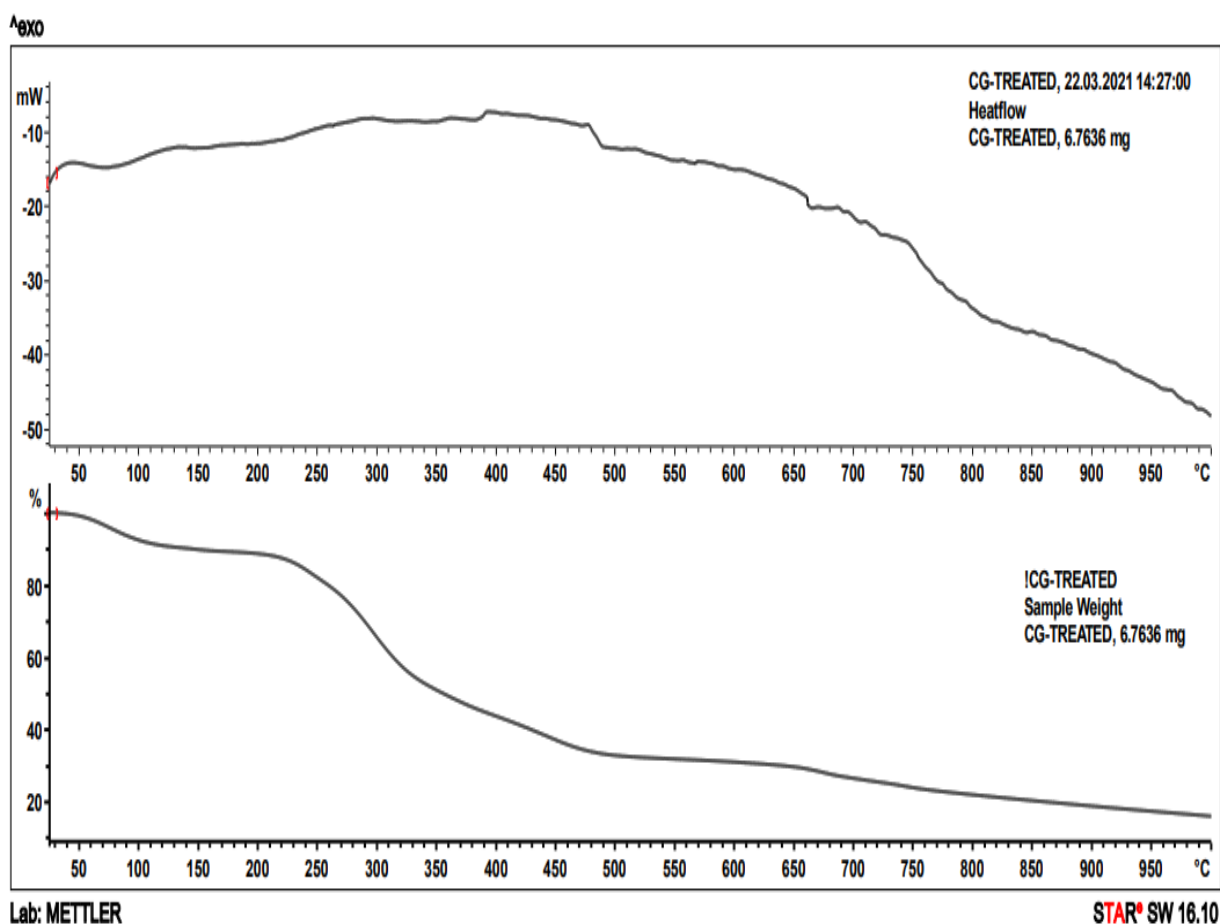


Fig:5.8 : Treated CGF TGA

CHAPTER-6

SUMMARY AND CONCLUSION

6.1 SUMMARY

The CGF fiber tensile test results shown us depending upon the critical gauge length 10mm fiber performs better for composite preparation with tensile strength of 304.169 MPa. The chemical analysis of the fiber has shown us a substantial increase in ash, cellulose, pectin. whereas wax, moisture, hemicellulose and lignin of the fibre decreased after alkali treatment. The chemical compositions of cellulose, ash, and pectin after alkali treatment are 70.96% to 73.42%, 2.19% to 2.74%, and 5.38% to 6.38% this helps in betterment of composite preparation. High cellulose, lignin, wax, and moisture chemical composition decreased to 28.74% to 17.68%, 12.19% to 11.95%, 0.52% to 0.44%, and 8.48% to 5.54%. Surface morphology of the CGF represents wax, lignin, pectin, and impurities on the surface of the sodium carbonate treated fiber produced an improvement in roughness on the surface. Tensile strength for 15% sodium carbonate treated CGF is 341.295 MPa, it is 12% more than untreated fiber whereas there was 8% decrease between 5% and 10% alkali treated fiber. FTIR results supports the chemical analysis by representing broad peaks at 3289 cm^{-1} and 2905 cm^{-1} has CH stretching (cellulose) responsible for this result. The peak (21.6°) of sodium carbonate-treated CGF was noticeably higher than untreated CGF, indicating that the sodium carbonate solution penetrated the fiber and dissolved low molecular content. Heat treatment between 290°C to 355°C shows highest drop of 50% mass reduction, indicates the removal or degraded hemicellulose content and glycosidic links of cellulose.

6.2 CONCLUSION

Based on the discussions made from the obtained results, the mechanical properties of untreated and alkali treated *coccinia grandis* is known. From the chemical analysis the major impacts of the alkali on the fibre is differentiated. Thermal stability of the fiber is observed in TGA. Fiber bonding and chemical properties are observed in FTIR. Surface morphology and fiber structure are represented by SEM and XRD.

REFERENCES

- [1] Asma El Oudiani Ben Sghaier, Yassin Chaabouni, Slah Msahli, Faouzi Sakli, Morphological and crystalline characterization of NaOH and NaOCl treated Agave americana L. fiber, Industrial Crops and Products, Volume 36, , March 2012, Issue 1 Pages 257-266. <https://doi.org/10.1016/j.indcrop.2011.09.012>
- [2] Agu, OU Njoku, FC Chilaka, D Agbiogwu, KV Iloabuchi, B Ukazu, Physicochemical properties of lignocellulosic biofibres from South Eastern Nigeria: Their suitability for biocomposite technology, AJOL volume 13, no 20, 2014. DOI: 10.5897/AJB2013.13443
- [3] Abraham. A. P, Mohammed.M. P, Ajith Kumar.S.C, George.S, Thomas, Thermoanalytical Techniques of Nanomaterials, in: Characterization of Nanomaterials, Elsevier, 2018: pages. 213–236. <https://doi.org/10.1016/B978-0-08-101973-3.00008-0>
- [4] Baley.C ,Analysis of the flax fibres tensile behaviour and analysis of the tensile stiffness increase, Composites Part A: Applied Science and Manufacturing ,Volume 33, Issue 7, 1 July 2002, Pages 939-948. [https://doi.org/10.1016/S1359-835X\(02\)00040-4](https://doi.org/10.1016/S1359-835X(02)00040-4)
- [5] Bibin Mathew Cherian, laly a. pothan, Tham nguyen-chung, gu`nter mennig, kottaisamy .m, and Sabu Thomas, A Novel Method for the Synthesis of Cellulose Nanofibril Whiskers from Banana Fibers and Characterization, J. Agric. Food Chem., volume 56, issue 14,2008, pages 5617–5627. <https://doi.org/10.1021/jf8003674>

- [6] Brigida.A.I. S, Calado.V.M. A, Gonçalves.L.R. B, Coelho.M.A. Z, Effect of chemical treatments on properties of green coconut fiber, Carbohydrate Polymers, volume-79, issue-4, March 2010,832–838. <https://doi.org/10.1016/j.carbpol.2009.10.005>
- [7] Bunaciu, Andrei A., Elena gabriela Udriștioiu, and Hassan Y. Aboul-Enein. “X-Ray Diffraction: Instrumentation and Applications.” Critical Reviews in Analytical Chemistry volume-45, issue. 4, October 2, 2015:pages 289–99. <https://doi.org/10.1080/10408347.2014.949616>
- [8] Balla.V. K, Kate.K.H, Satyavolu.j, Singh.P, Tadimetri.J.G. D, Additive manufacturing of natural fiber reinforced polymer composites: Processing and prospects, Composites Part B: Engineering. Volume -174 ,2019. 106956. <https://doi.org/10.1016/j.compositesb.2019.106956>
- [9] D.-C. Li, H. Jiang, the thermochemical conversion of non-lignocellulosic biomass to form biochar: A review on characterizations and mechanism elucidation, Bioresource Technology. Volume - 246, 2017, pages 57–68. <https://doi.org/10.1016/j.biortech.2017.07.029>.
- [10] Donaldson Lloyd, Microfibril Angle: Measurement, Variation and Relationships – A Review, IAWA Journal, Volume-29, Issue-4, 2008, Pages 345–386, <https://doi.org/10.1163/22941932-90000192>
- [11] Fiore.V, Scalici.T, Di Bella.G, & Valenza.A, A review on basalt fibre and its composites. Composites Part B: Engineering, volume 74, 2015, pages 74–94. doi:10.1016/j.compositesb.2014.12.034

- [12] Garette Jebadurai.S, Edwin Raj.R, Sreenivasan.V.S, Binoj, Comprehensive.J.S characterization of natural cellulosic fiber from *Coccinia grandis* stem, Carbohydrate Polymers ,Volume 207, 1 March 2019, Pages 675-683. <https://doi.org/10.1016/j.carbpol.2018.12.027>
- [13] Heru Suryanto, Eko Marsyahyo, Yudy Surya Irawan & Rudy Soenoko (2014) Morphology, Structure, and Mechanical Properties of Natural Cellulose Fiber from Mendong Grass (*Fimbristylis globulosa*), Journal of Natural Fibers, volume 11, issue 4, pages 333-351. DOI: 10.1080/15440478.2013.879087
- [14] Houcem Nailia, Ahmed Jelidia, Oualid Limama, Ramzi Khiari, Extraction process optimization of *Juncus* plant fibers for its use in a green composite, Industrial Crops and Products, Volume 107, issue 15, November 2017, Pages 172-183. <https://doi.org/10.1016/j.indcrop.2017.05.006>
- [15] Harwood.J, Harwood.R, Testing of natural textile Fibres, Volume 1 in Woodhead Publishing Series in Textiles, 2012, Pages 345-390. <https://doi.org/10.1533/9780857095503.1.345>
- [16] Indran.S, Edwin Raj.R, Characterization of new natural cellulosic fiber from *Cissus quadrangularis* stem, Carbohydrate Polymers, Volume 207, 1 March 2019, Pages 675-683. <https://doi.org/10.1016/j.carbpol.2018.12.027>
- [17] Indran, S., & Raj, R. *Characterization of new natural cellulosic fiber from Cissus quadrangularis stem. Carbohydrate Polymers, volume 117, 2015, Pages 392–399.* doi:10.1016/j.carbpol.2014.09.072

- [18] Jayaramudu.J, Guduri.B. R, & Varada Rajulu.A, Characterization of new natural cellulosic fabric *Grewia tilifolia*. *Carbohydrate Polymers*, volume 79, issue 4, pages 847–851. doi:10.1016/j.carbpol.2009.10.046
- [19] Kabir.MM, Wang.H, Lau.K. T, and Cardona.F, Chemical treatments on plant-based natural fibre reinforced polymer composites: An overview *Composites Part B: Engineering*, Volume 43, Issue 7, October 2012, Pages 2883-2892. <https://doi.org/10.1016/j.compositesb.2012.04.053>
- [20] Liu.D, Han.G, Huang.J, & Zhang.Y, Composition and structure study of natural *Nelumbo nucifera* fiber. *Carbohydrate Polymers*, volume 75, issue 1, pages 39–43. doi:10.1016/j.carbpol.2008.06.003
- [21] Muhammad Ramzan Abdul Karim, Danish Tahir, Azhar Hussain, Ehsan Ul Haq & Khurram Imran Khan, Sodium carbonate treatment of fibres to improve mechanical and water absorption characteristics of short bamboo natural fibres reinforced polyester composite, *Plastics, Rubber and Composites*, Volume-49, Issue-10, 2020. <https://doi.org/10.1080/14658011.2020.1768336>
- [22] Muhammad H. Hamidon, Mohamed T.H. Sultan, Ahmad H. Ariffin, Ain U.M. Shah, Effects of fibre treatment on mechanical properties of kenaf fibre reinforced composites: a review, *Journal of Materials Research and Technology*, Volume 8, Issue 3, May–June 2019, Pages 3327-3337. <https://doi.org/10.1016/j.jmrt.2019.04.012>
- [23] Na Lu , Subhashini Oza , Thermal stability and thermo-mechanical properties of hemp-high density polyethylene composites: Effect of two different chemical modifications, *Composites: Part B*, Volume 44, Issue 1, January 2013, Pages 484-490. <https://doi.org/10.1016/j.compositesb.2012.03.024>

- [24] Paul Wambua, Janlvens, Ignaz Verpoest, Natural fibres: can they replace glass in fibre reinforced plastics, Volume-63, Issue-9, 2003, Pages :1259-1264. [https://doi.org/10.1016/S0266-3538\(03\)00096-4](https://doi.org/10.1016/S0266-3538(03)00096-4)
- [25] Rahaman MN, Hossain MS, Razzak M, Muhammad B Uddin, A M sarwaruddin Chaudary, Rahual A khan, Effect of dye and temperature on the physico-mechanical properties of jute/PP and jute/LLDPE based composites, Heliyon, Volume 5, Issue-6, 2019. <https://doi.org/10.1016/j.heliyon.2019.e01753>
- [26] ROWELL.R.M, Natural fibres: types and properties, Properties and Performance of Natural-Fibre Composites, Woodhead Publishing , 2008, Pages 3-66. <https://doi.org/10.1533/9781845694593.1.3>
- [27] Senthamarai kannan.P, Kathiresan.M, Characterization of raw and alkali treated new natural cellulosic fiber from Cocciniagrandsis.L Carbohydrate Polymers, Volume 186, 15 April 2018, Pages 332-343. <https://doi.org/10.1016/j.carbpol.2018.01.072>
- [28] Suyambulingam Indran, Robinson Dhas Edwin Raj, Brainerd Samuel Sundar Daniel, Joseph Selvi Bino, Comprehensive Characterization of Natural Cissus quadrangularis Stem Fiber Composites as an Alternate for Conventional FRP Composites, J Bionic Eng, volume 15 2018, pages 914–923. DOI: <https://doi.org/10.1007/s42235-018-0078-9>
- [29] Senthamarai kannan.P, Saravanakumar S.S, Arthanarieswaran.V.P & Sugumaran.P (2015): Physicochemical Properties of New Cellulosic Fibers From Bark of Acacia Planifrons, International Journal of Polymer Analysis and Characterization, Volume 21, Issue 3, 2016 DOI: 10.1080/1023666X.2016.1133138
- [30] Saravanakumar.S. S, Kumaravel.A, Nagarajan.T & Ganesh Moorthy.I, Investigation of Physico-Chemical Properties of Alkali-Treated Prosopis juliflora

Fibers, International Journal of Polymer Analysis and Characterization, volume 19, issue 4,2014, pages 309-317. DOI: 10.1080/1023666X.2014.902527

- [31] Shakeri. A and Raghimi M, Studies on mechanical performance and water absorption of recycled newspaper/glass fiber reinforced polypropylene hybrid composites, JReinfPlast Compos, Volume- 29, Issue-7, 2010, Pages: 994-1005. <https://doi.org/10.1177/0731684408102700>

- [32] Velmurugan.R, Manikandan.V, Mechanical properties of palmyra/glass fiber hybrid composites. Composites, Part A, Volume-38, Issue-10,2007, Pages:2216-2226, <https://doi.org/10.1016/j.compositesa.2007.06.006>

- [33] XueLi, Lope G.Tabil & Satyanarayan Panigrahi chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review Journal of Polymers and the Environment volume 15, 04 January 2007, pages 25–33. DOI: 10.1007/s10924-006-0042-3