

# **DEVELOPMENT AND CHARACTERIZATION OF NEW NATURAL FIBER**

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Engineering degree in  
Mechanical Engineering

By

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**SCHOOL OF MECHANICAL ENGINEERING**

**SATHYABAMA**

**INSTITUTE OF SCIENCE AND TECHNOLOGY**

**(DEEMED TO BE UNIVERSITY)**

Accredited with grade "A++" by NAAC

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of VELISALA KARTHIK (Reg. No. 40150047) and CHIKKUDU VENKATA VIJAY KUMAR (Reg. No. 40150003) who carried out the project entitled "DEVELOPMENT AND CHARACTERIZATION OF NEW NATURAL FIBER" under my supervision from December 2023 to April 2024.

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## DECLARATION

We VELISALA KARTHIK (Reg. No. 40150047) and CHIKKUDU VENKATA VIJAY KUMAR (Reg. No. 40150003) hereby declare that the Project Report entitled "**DEVELOPMENT AND CHARACTERIZATION OF NEW NATURAL FIBER**" done by us under the guidance of Dr. R. SIVA., M.E., Ph.D., is submitted in partial fulfilment of the requirements for the award of Bachelor of Engineering degree in Mechanical Engineering.

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## ABSTRACT

The environmental threats posed by manmade composites during their disposal and depletion of fossil fuel resources have necessitated the application of ecofriendly materials for composite making. In the recent decades, many plant fibers with promising potential required for reinforcing composites have been identified and used for various applications. In this paper, the extraction and characterization of fiber derived from *Grewia villosa* bark was discussed. The fiber has a density of 1.21 g/cm<sup>3</sup>, lower than synthetic and natural fibers, with a high cellulose content of 65.16%. It exhibits an average tensile strength of 160.74±107.02 MPa and a Young's modulus of 6.02±3.89 GPa, offering mechanical robustness. With a thermal stability of 219.32°C, surpassing some natural fibers, it's suitable for temperatures below 200°C. Its 49% crystallinity and large crystal size of 27.28 nm enhance mechanical strength and reduce reactivity with chemicals and vapor. SEM images show a lack of porosity and a rough surface, making it an excellent reinforcement option for composites with low strength and high thermal stability.

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## LIST OF SYMBOLS AND ABBEREVIATIONS

|      |                                         |
|------|-----------------------------------------|
| NaOH | Sodium Hydroxide                        |
| FSP  | Fragrant Screw Pine                     |
| GPa  | Giga Pascal                             |
| MPa  | Mega Pascal                             |
| °C   | Degree Celsius                          |
| CTF  | Corypha Taliera Fruit                   |
| NBPF | Natural Bamboo Pulp Fibers              |
| AC   | Acacia Concinna                         |
| ALF  | Acacia Leucophloea Fiber                |
| CPF  | Carica Papaya Fiber                     |
| FRF  | Ficus Racemosa Fiber                    |
| PJF  | Prosopis Juliflora Fiber                |
| GVF  | Grewia Villosa Fiber                    |
| FTIR | Fourier Transform Infrared Spectroscopy |
| TGA  | Thermogravimetric Analysis              |
| DTG  | Differential Thermogravimetry           |
| DTA  | Differential Thermal Analysis           |
| SEM  | Scanning Electron Microscopy            |
| XRD  | X-Ray Diffraction                       |
| KAE  | Kinetic Activation Energy               |
| CI   | Crystallinity Index                     |
| CS   | Crystalline Size                        |
| μm   | Micro Meter                             |
| Nm   | Nano Meter                              |
| Wt%  | Weight Percentage                       |

# CHAPTER 1

## INTRODUCTION

For centuries, natural fibers generated from plants and animals have served as the foundation of human civilization, providing us with clothes and other essential necessities. However, the introduction of synthetic fibers in the twentieth century resulted in a significant change in the textile business. These synthetic fibers derived from petrochemicals provided durability, flexibility, and moisture resistance, resulting in broad use. However, this transition has not been without costs.

Synthetic fibers, while useful in some applications, provide considerable environmental issues. Synthetic fiber manufacturing is energy-intensive and significantly reliant on nonrenewable resources, which contributes to the depletion of fossil fuel reserves. Furthermore, the production process generates a significant quantity of greenhouse emissions. Synthetic fibers can contribute to water pollution by releasing toxic substances like heavy metals and formaldehyde into streams and soil, which can cause respiratory and cardiovascular problems. Furthermore, synthetic microfibers are a major cause of microplastic contamination, especially in marine habitats, where they can be consumed by aquatic species and enter the food chain.

Natural fibers, on the other hand, provide several advantages for the environment and human health. They require a lot less energy to produce and are biodegradable and renewable. In addition to being breathable, comfortable, and capable of retaining moisture, natural fibers are perfect for use in apparel and other consumer goods. Customers have a healthier option because they are less prone to experience allergic reactions or skin discomfort. In addition, compared to synthetic fibers, natural fibers have a reduced carbon footprint since they produce fewer greenhouse gases during production and processing.

The growing trend of substituting natural fibers for synthetic ones is to make products more eco-friendly and sustainable. Natural fibers offer equivalent, if not better, performance attributes and are utilized in multiple industries, including

construction, automotive, and aerospace. For example, natural fibers have demonstrated encouraging results in the reinforcement of geopolymer matrices, offering an ecological advantage over synthetic fibers. Natural fibers are becoming a more feasible replacement to man-made fibers in many applications due to their adaptability and technological developments in processing.

## 1.1 NATURAL FIBERS

The phrase 'natural fiber' implies fibres derived from flora and fauna. These fibers have several uses in the creation of composite materials. Natural fibers have been utilized by mankind for thousands of years for making clothes, housing, and other necessities. These fibers are renowned for their toughness, biodegradability, and renewability. They are also thought to be more ecologically friendly than artificial fibers, which are manufactured from petroleum sources. Natural fibers have a wide range of uses, including garments, household textiles, and commercial textiles.

## 1.2 HISTORY OF NATURAL FIBERS

The history of natural fibers is a fascinating journey through time, tracing the integral role these materials have played in human civilization. From prehistoric times to modernity, natural fibers have been essential for clothing, shelter, and countless other applications.

Early humans used plant fibers such as flax, cotton, and hemp to create textiles for clothing and shelter. These fibers were readily available and easy to manipulate. In ancient Egypt, for example, linen made from flax was highly prized and used for mummification and clothing.

Silk, derived from the cocoons of silkworms, emerged as a luxurious fiber in China around 2700 BCE. The Silk Road, a network of trade routes, facilitated the exchange of silk and other goods between China and the West, shaping cultures and economies along the way.

During the Middle Ages, Europe became a center for wool production, with sheep grazing on the lush pastures. Woolen textiles, such as those woven in Flanders and England, took a vital role in the region's economy and culture.

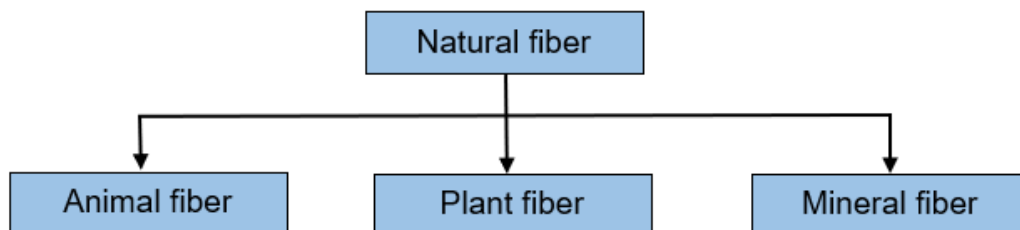
The discovery of the New World in the 15th century introduced Europe to new natural fibers like cotton and jute. Cotton, in particular, became a staple in the textile industry and played a pivotal role in the growth of the American economy through plantations.

In the 19th century, the Industrial Revolution brought innovations like cotton gin and mechanized looms, dramatically increasing textile production. However, this period also saw the exploitation of natural resources and labor in the name of industrial progress.

Today, natural fibers remain indispensable in various industries, including fashion, agriculture, and construction. Innovations continue to explore sustainable and eco-friendly uses for these historical materials, emphasizing the importance of balancing tradition with modernity in our quest for a more sustainable future.

### 1.3 CLASSIFICATION OF NATURAL FIBERS

Categorization of natural fibers was done based on the fibers source and composition. Figure 1.1 depicts the categories of natural fibers.



***Fig 1.1: Classification of Natural Fibers***

#### ***1.3.1 Animal Fibers***

Animal fibers are sourced from animals and are known for their warmth and softness. Wool is one of the most popular animal fibers, derived from the fleece of sheep. Mohair comes from the Angora goat and is renowned for its lustrous appearance. Silk is another exquisite animal fiber produced by silkworms. These fibers are biodegradable and have excellent insulation properties, making them ideal for cold-weather clothing and textiles.

### **1.3.2 Plant fibers**

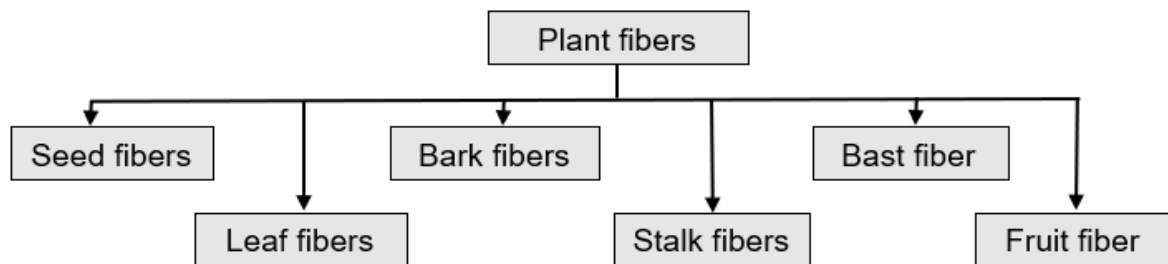
Plant fibers are plant-based and primarily composed of cellulose, a complex carbohydrate. Cotton is the most well-known cellulose fiber, valued for its breathability, absorbency, and comfort. Linen, extracted from flax plants, offers exceptional strength and cooling properties, making it suitable for summer apparel. Bamboo fibers are gaining popularity due to their eco-friendliness and softness.

### **1.3.3 Mineral Fibers**

These types of fibers are extracted from the minerals. The most notable mineral fiber is asbestos, which was once widely used for its heat resistance and durability. However, due to health concerns associated with asbestos exposure, its use has diminished significantly.

## **1.4 PLANT FIBERS**

Fibers are fine thread-like materials. The fibers that are derived from the different parts of plants are called as plant fibers. Few examples of plant fibers are cotton, jute, sisal and hemp.



**Fig 1.2: Types of Plant Fibers**

The plant fibers are sorted into different types and the figure 1.2 shows the types of plant fibers.

- Seed fibers – seeds are used as source to collect fibers in these fibers type.
- Leaf fibers – in this type of fibers, leaves of the plants are the source for fiber. Pineapple and banana leaf fibers are such types of fibers.

- Fruit fibers – fibers that are derived from the tree fruits. (Coconut fiber, for example).
- Bark fibers – these types of fibers are derived from the tree bark.
- Stalk fibers – the source of the fiber was the stalk of a plant. Few of them are bamboo fibers and wheat straws.
- Bast fibers – fibers which are derived from the exterior layer of stem. Few of them are vine, hemp, jute and rattan fibers.

### 1.5 BARK FIBERS

Bark fibers are a type of plant fiber that are obtained from the inner bark of certain dicotyledonous plants. They are known for their strength and durability and have been used for centuries to make a variety of items, including clothing, baskets, and paper.

The process of making bark fibers is labor-intensive and requires a great deal of skill and patience. However, the resulting fibers are strong, durable, and have a unique texture that makes them ideal for use in multiple applications.

One of the most important characteristics of bark fibers is their strength. Bark fibers are known for their ability to withstand wear and tear, making them ideal for use in items that need to be strong and durable. Additionally, bark fibers have a unique texture that makes them ideal for use in clothing and other textiles. The texture of bark fibers is rough and slightly textured, which gives them a distinctive look and feel.

Bark fibers have many applications in the textile industry. They can be utilized to make multiple items which includes clothing, bags, baskets, and paper. Additionally, bark fibers can be used to make rope and twine. Bark fibers also have their role in the construction industry to make insulation and other building materials.

## CHAPTER 2

### LITERATURE SURVEY

(Wang et al., 2007) This study focused on how the level of individualization affects the morphology of hemp fibers. Nanofibers consist of bundles of cellulose fibers estimated to be 30–100 nm wide and several micrometers long. Through the application of chemical treatments, the hemp microfibers were individualized and a network structure of hemp nanofibers was formed through mechanical processes. Chemical analysis disclosed that the particular chemical treatment elevated the percentage of cellulose from 75% to 94%. Subsequent bleaching helped purify the cellulose. FTIR analysis revealed that pectin was fractionally deducted during the individualization process XRD analysis revealed that the relative crystallinity of the fibers elevated after every step of chemical and mechanical treatment. Notably, the relative crystallinity of the hemp fibers incremented after each chemical treatment step, with the nanofibers exhibiting 71% crystallinity compared to 57% for the untreated fibers.

(Adugna Ayalew & Fenta Wodage, 2022) Sisal fibers have considerable strength and are suitable for use as carrier materials for polymers. The fibers are treated with 2%, 6% and 10% of NaOH for a time period of 24, 48, and 72 h, respectively. The treated and untreated fibers functional groups are studied by FTIR. Thermal stability tests were performed on sisal fibers using thermogravimetric analysis (TGA). FT-IR studies confirmed the degradation of lignin and hemicellulose, and the highest intensity could decrease with alkali content, indicating the deficiency of hemicellulose. In between the temperature 59 °C to 378 °C, a large mass loss of sisal fibers is observed, which correlates with the removal of surface water. The strong endothermic peak at 59 °C in his TGA sheets of sisal fibers indicates the dehydration of the outer surface of the fibers and the influence of the interlayer matrix. The surface-treated sisal fibers showed improved resistance to temperatures below 260 °C.

(Cecci et al., 2020) This study focuses on finding eco-friendly applications for banana pseudostem fibers. The morphological properties of cellulose fibers made out of pseudo stem allow the production of materials with robust mechanical

properties. After mechanical extraction of fibers from banana pseudo stem, the diameter, tensile strength, and elongation at break were analyzed by tensile tests. Furthermore, we used scanning electron microscopy to examine their morphological structures. Irrespective of the FTIR absorption bands and chemical composition, the single fiber tensile strength test proved that the fiber has high tensile strength of 590 MPa which is greater than jute (249 MPa). The remarkable mechanical strength of banana pseudostem fibers can be attributed to elevate crystallinity (approximately 67%) and its unique morphology, as determined from FTIR spectra. These fibers have shown to be the strongest among the natural fibers, making them excellent not only for reinforcing composite materials, but can also be utilized for applications such as packaging, clothes, carpets, ropes, and even decoration and design.

(Selvan et al., 2022) ) Biodegradable natural plant fibers derived from plant buttress roots are widely explored as an alternate to artificial fibers in lightweight composite applications which are bio-based. To estimate the ability of FSP fibers as unique reinforcement materials multiple characterization tests were done. It was known that the density was low ( $1.3852 \text{ g/cm}^3$ ) and diameter was found to be 27-280  $\mu\text{m}$ . The cellulose (73.10 wt%) which was quite high and minimal wax (0.35 wt%) of FSP fibers contribute to its good specific strength and bonding properties. The raw FSP fibers have a tensile strength of  $915 \pm 195 \text{ MPa}$ , a Young's modulus of  $33 \pm 12 \text{ GPa}$ , and environmental standards.

(Tamanna et al., 2021) This research focuses on identifying novel natural fibers derived from the *Corypha taliera* fruit (CTF) to offer a potential eco-friendly alternative to harmful synthetic fibers. The study involved a detailed investigation of multiple characteristics of CTF fibers. Results showed cellulose content (55.1 wt%) as well as crystallinity (62.5%) in CTF fibers through XRD and chemical composition analysis. Multiple functional groups such as cellulose, lignin, and hemicellulose within the fiber was recognized by FTIR analysis. The CTF's average diameter, specific gravity and linear density were around 131  $\mu\text{m}$ ,  $0.86 \text{ g/cm}^3$  and 43 tex respectively. The CTF fiber exhibited a maximum tensile strength of 53.55 MPa and a Young's modulus of 572.21 MPa. The thermal analysis indicated the fiber's thermal sustainability up to  $230^\circ\text{C}$ , affirming CTF as a capable reinforcement material in composite materials with fibers as reinforcement.

(do Nascimento et al., 2021) The research conducted focused on lignocellulosic fibers derived from *Yucca aloifolia* L. leaves, and its characterization. The crystallinity of cellulose was determined to be 69.43%, and the fibers had a good cellulose content of 52.5% and thermally stable up to 193.4 °C. Single fiber tensile tests showcased an average tensile strength of 800 MPa, a Young's modulus of 39 GPa, and 2% strain at failure. SEM test showed the appearance of numerous parenchymal cells and non-cellulosic components on the fiber surface, indicating the possible utilization of *Yucca aloifolia* L. fibers as fibrous reinforcement material in polymeric matrices.

(Khan et al., 2022) The *Citrullus lanatus* climber fibers were undergone multiple characterization studies. Test results unveiled a cellulose content of 53.7 wt. %, a density of 1227 kg/m<sup>3</sup>, and a CI of 33.33% in *Citrullus Lantau's* climber fibers, suggesting their potential use as reinforcement for composites which requires less weight. SEM analysis revealed the hollow structure of the fiber cross-section and a rough surface, which enhances the bonding mechanism in biocomposite manufacturing. The TGA showed the thermal stability of *Citrullus lanatus* climber fibers up to 325°C, while the ultimate tensile strength was recorded at  $38.1 \pm 1.56$  MPa.

(Manimaran et al., 2020) The current study presents an investigation into the characteristics of natural bamboo pulp fibers (NBPF) encompassing physical, chemical, morphological and thermal properties. The NBPF displays a diameter of approximately 26 micrometers and a density of around 972 kilograms per cubic meter. The tensile strength of a single fiber is observed to be about  $65.51 \pm 1.15$  MPa with a Young's modulus of roughly  $49.5 \pm 1.31$  GPa. Noteworthy is the cellulose content of NBPF at 73.20 wt %, indicating a superior cellulose composition compared to other similar fibers. XRD analysis reveals a CI of about 53.3% and a CS of around 4.72 nm. The thermal gravimetric analysis (TGA) highlights the NBPF's resilience, enduring temperatures up to 356°C with a 50% cut off in mass. These findings underscore NBPF's potential suitability for creating polymer composites utilized in automobile and construction materials which need to be light in weight. Additionally, FTIR confirm the footprints of varied chemical compositions and respective functional groups. AFM unveils the rough fiber surface.

(Amutha & Senthilkumar, 2021) The goal of the research was to find the viability of fibers obtained from the *Acacia concinna* (AC) plant bark for reinforcing composites. A thorough chemical analysis reveals that AC fibers contain around 59.43 wt. % cellulose and 12.78 wt. % hemicellulose. Utilizing FTIR testing, various chemical compositions and corresponding functional groups within the AC fibers are identified. XRD analysis pinpoints a CI of approximately 27.5% and a crystalline size of about 4.17 nm. Noteworthy is the AC fiber's superior thermal stability up to 225°C, reaching a ultimate degradation temperature of 326°C with a kinetic activation energy of approximately 69.33 KJ/mol and a lower density of 1365 kg/m<sup>3</sup>, making it a desirable reinforcement material in polymer matrices. SEM and AFM studies reveals key roughness parameters.

(Arthanarieswaran et al., 2015) Fibers are extracted from the the *Acacia leucophloea* bark. Various tests were done to find the suitability of fiber. Significant findings include a elevated cellulose content of 68.09 wt.%, a density of 1385 kg/m<sup>3</sup>, a CI of 51%, tensile strength ranging from 317 to 1608 MPa, and Young's modulus varying from 8.41 to 69.61 GPa. Thermal analyses indicate that ALFs degrade around 220°C with a kinetic activation energy of 73.1 kJ/mol. The predominant cellulose content within ALFs offers enhanced strength among bark fibers, while their low density positions them as ideal components for lightweight composite materials.

(Senthamarai Kannan, Sanjay, et al., 2019) New natural fibers are sourced from the bark of *Alum tree*. The fiber study unveiled the cellulose, lignin and hemicellulose percentage as 65.4%, 15.61% and 14.32% respectively. CI was detected as 63.78%. TGA study revealed that AAF has thermal stability up to 330.6 °C, while FTIR analysis identified its functional groups. AAF exhibited a tensile strength of 640 ± 13.4 MPa and a strain rate of 1.57 ± 0.04%. This study indicates that AAF may be applicable as a reinforcing agent in composites for various applications. AAF has a high cellulose content and low microfibril angle, which ensures a relatively high tensile strength of the fibers. From the above results, it can be proposed to use AAF as a reinforcing material for composite materials for multiple applications which include panels for furniture, doors and even in bricks.

(Gopinath et al., 2022) Physical, mechanical, thermal properties and chemical characteristics were obtained from the bark of Album tree (AS) are reported. Prominent cellulose and lignin content was obtained (60.76% and 14.64%), which significantly contributed to good mechanical properties of AS fibers. FT-IR and XRD studies stated high crystallinity (57.69%). The thermal stability of ASF obtained from thermogravimetry, thermogravimetry, and differential calorimetry curves was studied, and the maximum decomposition temperature of ASF was 306.19 °C, and the kinetic activation energy (KAE) was 64.01 kJ/mol. I understand that. It became clear. The tensile strength and tensile elongation of ASF were 381–1092 MPa and 1.65–4.42%, respectively, and the tensile modulus values were 9.68–42.31 GPa. From this result, it can be concluded that ASF is suitable for the production of green composites as it possesses the essential properties required for reinforced composites.

(Saravana Kumaar et al., 2019) The current research discusses the properties of Carica papaya fibers (CPFs) sourced from the bark of papaya plant. The chemical compositions of CPFs were defined and determined using conventional procedures. The physiochemical parameters of CPFs, including 56.34% crystallinity, low cellulose content of 38.71%, 11.8% of hemicellulose, and 943 kg/m<sup>3</sup>, were contrasted to those of comparable natural fibers. The thermal degradation pattern predicted using TGA and DTG curves, as well as the thermal characteristics of CPFs, illustrate that cellulose is stable below 322.1°C. FT-IR and X-ray studies revealed that the CPFs comprised a high cellulose content and CI of 56.34%. The topographic characteristics of CPFs demonstrated that surface modification is necessary to improve the surface properties. The findings indicate that CPFs might be employed as a possible reinforcement in a polymer matrix composite construction.

(Kumar et al., 2018) This study describes the procedure of extraction and validation of new fibers from the C. pentandra plant bark with the intention to find their possible application as reinforcement for matrix. Natural fibers are the most promising replacement to synthetic fibers due to their less ratio value of weight-strength and biodegradable properties. They have additionally been used as reinforcing materials in polymer matrix composites. CPFs include 60.9% cellulose (w/w), 17.5% hemicellulose, and 23.5% lignin. Moreover, the density of the fiber was minimal (682

kg/m<sup>3</sup>) and CI 57.94%. Moreover, the resources of CPFs meant that they could potentially serve as a perfect substitute for artificial fibers in composites made of polymer matrix.

(Baskaran et al., 2018) The properties of *Dichrostachys Cinerea* (DCA) were investigated in this study. the fiber exhibited an cellulose content of 72.4 wt.%, 16.89 wt.% lignin and 13.08 wt.% of hemicellulose. The CI, density and tensile strength of the fiber are 57.82%, 1240 kg/m<sup>3</sup> and 873 ± 14 MPa, respectively. The mechanical characteristics of DCFs demonstrate that this was best alternatives for man-made fibers. Aside from the cellulose breakdown of DCFs at 359.3° as determined by TGA, chemical groups are detected using FTIR analysis. Finally, the validation results for DCFs clearly suggest the feasibility of reinforcement in polymer matrices.

(Manimaran, Saravanan, et al., 2021) In this study, fiber was resourced from *Ficus racemosa* bark. The cellulose content of FRF was 72.36% which was quite impressive with 10.45% of lignin. In addition to this, the density of the fiber was also low (895 kg/m<sup>3</sup>). In contrast to remaining natural fibers, FRFs have a high cellulose content (72.36 wt%). The FRFs appear to have minimal wax content (0.25%), which significantly increases bond strength between reinforcement and matrix. DTG indicates that the greatest deterioration peak temperature was reached at 310.56°C. FTIR analysis confirmed the existence of a chemical group in the fibers. The structure of the crystals was determined using XRD. The CI was determined as 57.32%. FRFs has tensile strength and young's modulus of 270.83 MPa and 67.45 GPa. Surface morphology was determined using SEM and AFM. The study showed that FRFs are appropriate to use as reinforcement in composites.

(Hossain et al., 2022) Retting technique was utilized for drawing out fiber from the jack tree bark. The fiber has been evaluated using conventional procedures. Fiber comprises of α-cellulose 79.32%, lignin 6.77%, hemicellulose 8.01%, ash 3.58% and extractives 2.32%. XRD testing indicates its high degree of crystallinity which is 86% and the microfibrillar angle of -29° was found. The FTIR measurement verifies the existence of the desired functional groups. TGA and DTG show high thermal stability with maximal degradation occurring at 358 °C for α-cellulose. The fiber is exhibited thermal stability up to 228°C. This breakdown behaviour suggests that the fiber is high in cellulose percentage and has crystalline structures. The fiber has a

density of 1.05 g/cc, which is smaller than many natural fibers which are known. The fiber's average strength and elongation were determined to be 2.6 g/denier and 5.9%, respectively. This fiber has moderate strength and good elongation capabilities. All of these qualities make this novel fiber suited for a wide range of advanced fibrous applications, including composite reinforcement, textiles, cellulose nanoparticles, activated or conductive carbon, biomaterials, and more.

(Saravanakumar et al., 2013) The present study focuses on the fibers extracted from *Prosopis juliflora* (PJF) which was till now not studied. PJF was thoroughly investigated to determine its chemical and physical characteristics. It had a greater lignin content (17.11%) and less density (580 kg/m<sup>3</sup>) than bark fibers from other plants. The free chemical compounds on it were investigated using FTIR and XRD. The single fiber tensile test of the fiber exposed that the fiber possesses  $558 \pm 13.4$  MPa tensile strength with  $1.77 \pm 0.04\%$  average strain rate and the microfibril angle was  $10.64^\circ \pm 0.45^\circ$ . Thermogravimetric examination showed the PJF thermal stability which was 217°C.

(Laifa et al., 2022) In this study, fibers are isolated from the *Silybum marianum* (SM) bark, with the goal of employing it as a possible reinforcement for composites. This study began with discussing fiber morphology and anatomy, as well as their physical, thermal, and mechanical characteristics. The fiber has 201.16 MPa tensile strength with Young's modulus of 15.97 GPa. XRD shows that the CI value was 45%. The maximum degradation temperature was found as 357.72°C and the fiber was thermally stable up to 250°C.

(Gopinath et al., 2023) The adaptability of a new cellulosic fiber derived from *Tithonia diversifolia* bark for a composite application was evaluated using a variety of characterization tests. *Tithonia diversifolia* fibers were examined using various characterization techniques and reported. The high cellulose content (63.9 wt.%) as well as lignin content (19.3 wt.%) of the TDFs make them more strong and stable. TDFs are better suited for green composites because to their lower density (1440 kg/m<sup>3</sup>), greater mechanical strength (243-386 MPa), and thermally stable (up to 337.17°C). The CI and CS of TDFs were evaluated via XRD to be 51.84% and 2.93 nm, respectively. The uneven surface of the fiber was detected using FESEM which allows enhanced bonding properties with polymer matrix.

## CHAPTER 3

### AIM AND SCOPE

#### 3.1 AIM OF THE PROJECT

The project named 'Development and Characterization of New Natural Fiber' aims to validate the potential of tree bark as a source of natural fiber for a variety of applications. This study seeks to collect and characterize fibers from *Grewia villosa* bark in order to establish their potential as a replacement to synthetic fibers. The characterization testing will involve measuring density and diameter, performing chemical analysis, and assessing mechanical qualities using single fiber tensile tests. Advanced analytical methods, including FTIR, XRD, TGA and SEM will be used to acquire knowledge into the fiber's structural and compositional properties. The ultimate objective is to compare these features to those of existing fibers, determining the feasibility of *Grewia villosa* bark fibers as a renewable and environmentally friendly material in a variety of businesses.

#### 3.2 SCOPE OF THE PROJECT

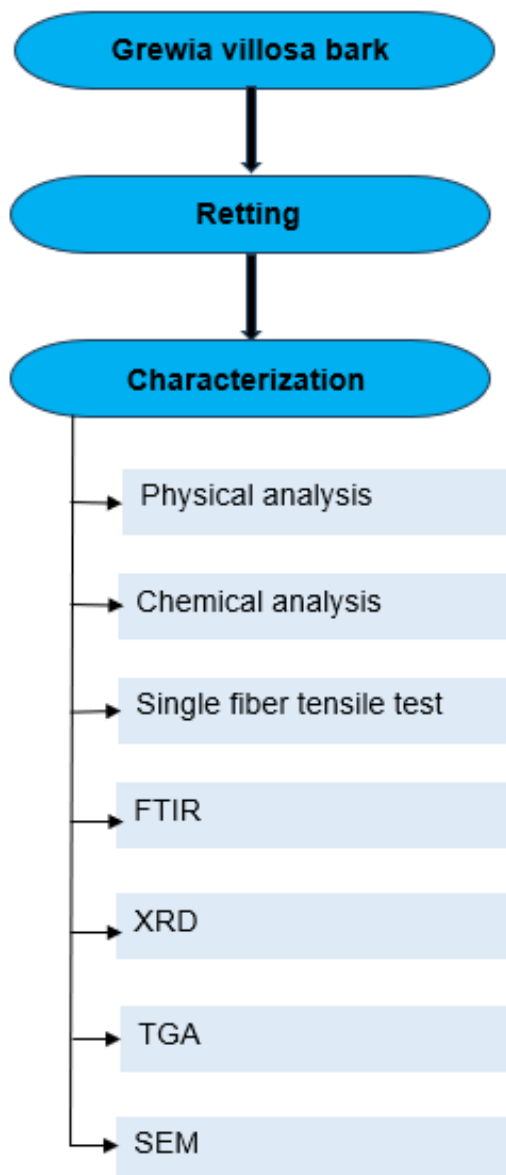
A comparison study will be done to contrast the newly produced fibers with current ones, with the goal of identifying areas where they may outperform or be more advantageous. The project's goal is to identify possible uses for these fibers in multiple sectors including composite materials, and insulation, which will contribute towards the creation of eco-friendly materials. The larger consequences include encouraging innovation in natural fiber research, supporting sustainable material use, and even influencing consumer preferences toward greener alternatives. This initiative might lead to substantial advances in material sciences and sustainable development.

## CHAPTER 4

### MATERIALS AND METHODS

#### 4.1 METHODOLOGY

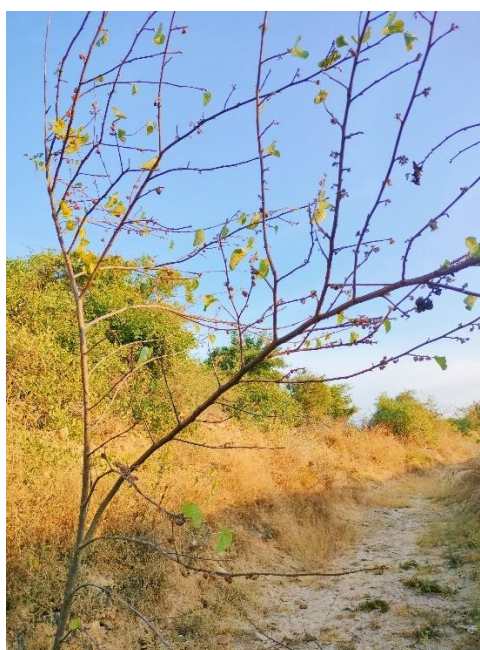
The figure 4.1 explains the methodology followed in the project. It starts with the derivation of the fiber from the *Grewia villosa* plant and characterization of fiber.



***Fig 4.1: Methodology***

## 4.2 MATERIALS

*Grewia villosa*, a climbing plant from the Malvaceae family, generally does not grow beyond 4 meters in height. Its leaves are sizeable, toothed, and heart-shaped. The flowers are yellowish-red/brown, growing in small groups, while the fruits, light red, are either whole or somewhat divided into four lobes, with a fibrous peel. This plant is gathered in the wild for various local purposes like food, medicinal use, and as a source of materials. Its natural habitat spans Tropical Africa, Arabia, Pakistan, East Indies, and India, typically thriving in highlands, lower valleys, beside roadsides, and at field edges. For this particular investigation, specimens of the plant were obtained from Paluguntipalli village in Prakasam district, Andhra Pradesh, India. Scientific research has shown that this plant exhibits biological properties such as antioxidants, anti-cancer effects, antibacterial and antimicrobial activities, as well as cytotoxic properties. Fatty acids and steroids are notably abundant in this plant.



**Fig 4.2: *Grewia Villosa* Tree**



***Fig 4.3: Grewia Villosa Bark***

#### 4.3 WATER RETTING

The tree's bark got separated from its branches. The bark received a gentle hammering to help water penetrate better. Subsequently, the bark was submerged in fresh water for about two weeks for microbial breakdown.



***Fig 4.4: Water Retting***

Following this, the fiber got manually extracted from the bark. The retrieved fiber underwent multiple rinses with tap water and then distilled water to eliminate impurities. Next, the cleaned fiber was left to dry under sunlight for a day to reduce moisture content. Further drying took place in a hot air oven to ensure complete moisture removal. The fiber was then packaged in an airtight container to ward off moisture until testing.



**Fig 4.5: Grewia Villosa Fiber**

#### 4.4 PHYSICAL ANALYSIS

The density of Grewia Villosa fiber was determined using a pycnometer filled with a known density liquid, methylbenzene (Toluene) ( $\rho_m = 866 \text{ kg/m}^3$ ). Equation 1 was applied to ascertain the fiber's density.

$$\rho_{\text{fiber}} = \left( \frac{m_2 - m_1}{(m_3 - m_1) - (m_4 - m_2)} \right) \rho_l \quad (1)$$

Where,

$m_1$  = Weight of an empty pycnometer in kilograms

$m_2$ = Weight of pycnometer filled with fiber in kilograms

$m_3$ = Weight of pycnometer filled with immersion liquid in kilograms

$m_4$ = Weight of pycnometer filled with a mixture of fiber and immersion liquid in kilograms

$\rho_l$ = Density of immersion liquid

$\rho_{\text{fiber}}$ = Density of fiber

#### 4.5 CHEMICAL ANALYSIS

Chemical analysis of natural fibers is a crucial process that helps in understanding their composition and properties. The natural fibers majorly comprise of cellulose, lignin, hemicellulose, wax, moisture content and ash. Various methods are applied to determine the chemical composition of natural fibers.

##### **4.5.1 Determination of cellulose**

Cellulose content of the natural fiber was evaluated through Kurschner and Hoffer's method. Initially, fibers are cleaned with distilled water and thoroughly dried in a hot air oven for 24 hours to eliminate any moisture. Subsequently, 150 mg of these dried fibers are subjected to hydrolysis in a mixture of 95% nitric acid and ethanol. The fibers are maintained in a hot air oven at 60°C until their weight remains constant, indicating the completion of the hydrolysis reaction. The remaining weight of the insoluble residue obtained after this process is a direct measure of the cellulose content within the fiber.

##### **4.5.2 Determination of hemicellulose**

The neutral detergent method is a technique used to evaluate hemicellulose content in AS fiber samples. It begins by refluxing the sample in a 10 ml solution comprising neutral detergent and sodium sulphite for an hour. A sintered glass crucible is then employed to separate the mixture, and residue is thoroughly washed with hot distilled water followed by ethanol solution. The remaining residue after these washes indicates the hemicellulose content within the fibers.

#### 4.5.3 Determination of lignin

Quantification of lignin content in the material followed the APPITA P11S-78 method, a standardized procedure widely used for this purpose. It commenced with the preparation of crushed fiber samples, each 300mg weight, which were subjected to hydrolysis using 72% sulfuric acid for an hour at a temperature of 30°C. Subsequently, samples of fiber which were treated are combined with a methylene chloride solvent solution and placed in an autoclave, where they were exposed to elevated temperatures at 125°C for a duration of 60 minutes. Following this process, the samples temperature was brought down to room temperature, after which the extracted lignin was meticulously refined. The equation given below was used to determine the lignin percentage.

$$\begin{aligned} &\% \text{ of insoluble lignin content} && (2) \\ &= \left( \frac{\text{weight of insoluble lignin}(g)}{\text{weight of fiber sample kept in oven}(g)} \right) \times 100 \end{aligned}$$

#### 4.5.4 Determination of wax

The quantification of wax content in the natural fibers was carried out using the meticulous Soxhlet extraction method, a well-established technique for extracting and measuring organic compounds. Initially, the fiber samples underwent a preparatory phase, involving thorough washing and subsequent drying in a hot air oven at 80°C for a period of 60 minutes. This ensured that the samples were free of any extraneous substances and moisture. The dried fibers were then meticulously chopped into uniform 10 mm-sized pieces and submerged in an ethanol solution for a prolonged period of 6 hours. This step allowed the wax content to dissolve into the ethanol from the fiber matrix. Following this, the wax-laden ethanol solution was segregated from the wet fibers by employing a segregating funnel containing chloroform. In the final stage of the process, the separator funnel was subjected to controlled heating, causing the chloroform to evaporate gradually. As a result, the wax, which had been dissolved in the chloroform, was left behind as a solid residue. By using below equation, we can calculate the wax percentage.

$$Wax(\%) = \left( \frac{(W_f - W_w)}{W_f} \right) \times 100 \quad (3)$$

Where,

$W_f$ = weight of dried fiber sample kept in oven (g)

$W_w$ = weight of wax extracted from the sample (g)

#### **4.5.5 Determination of ash**

The assessment of ash content in natural fibers adhered to the ASTM E 1755–01 standard, a well-established procedure. It involved subjecting a crushed fiber sample to complete incineration within a calorimeter set at a controlled temperature of 250°C. During this process, all organic matter in the fiber sample was consumed, leaving only the inorganic mineral residue in the form of ash. The precise quantity of this residual ash was then carefully measured and weighed.

#### **4.5.6 Determination of moisture content**

Moisture content in natural fibers was assessed using the conventional weight loss method. In this procedure, a 20 mg fiber sample was carefully placed inside an oven set at 100°C constant temperature. The sample was left undisturbed in the oven for of 4 hours and 30 minutes duration, allowing the moisture within the fibers to gradually evaporate. The process continued until a state of constant weight was achieved, indicating that all the moisture had been effectively removed. The percentage of moisture content was then calculated using below Equation:

$$Moisture\ content\ (\%) = \left( \frac{W_1 - W_2}{W_1} \right) \times 100 \quad (4)$$

Where,

$W_1$  = weight of the fiber before removing the moisture in grams

$W_2$  = weight of the fiber after removing the moisture in grams

#### 4.6 FOURIER TRANSFORM INFRA-RED SPECTROSCOPY (FTIR)

The fiber undergoes a series of steps in FTIR analysis. Initially, mortar and pestle was utilized to transform the fiber in to powder. This powder is then mixed with potassium bromide, creating a homogeneous mixture. To facilitate FTIR measurements, this mixture is compressed by using a hydraulic press into a pellet form, applying sufficient pressure for consolidation.

Once the pellet is prepared, it is exposed to infrared radiation. The FTIR spectrometer records the interaction between the radiation and the sample, generating a spectrum that displays peaks and valleys across the wavenumber range of  $4000\text{ cm}^{-1}$  to  $500\text{ cm}^{-1}$ . The scan rate is maintained at 32 scans per minute with a resolution of  $4\text{ cm}^{-1}$ .

#### 4.7 X-RAY DIFFRACTION (XRD)

X'pert Pro PANalytical system diffractometer was used to find the fiber crystallinity, crystal size and the structure of the molecules. The test was done over the range of  $2\theta$  from  $10^\circ$  to  $80^\circ$  with  $0.05^\circ$  step size. For the investigation, the generator parameters were tuned to 45 kV and 30mA with a scan rate of  $5^\circ/\text{min}$ . High-energy electrons were used to strike a copper target, creating a monochromatic stream of  $\text{CuK}\alpha$  radiation with 0.1542 nm wavelength. This beam was subsequently focused onto a sample on a revolving platform. The detector receives the diffracted radiation and counts the X-rays scattered by the sample. The assessment of the crystallinity index (CI), which implies the proportion of crystalline cellulose within the overall cellulose content comprising both crystalline as well as amorphous components, was conducted using the peak height technique. The CI value was determined by the equation below:

$$CI = \left( \frac{\text{Area of crystalline region}}{\text{Area of crystalline} + \text{amorphous region}} \right) \times 100 \quad (5)$$

Ability of fiber to absorb water and the chemical reactivity of the fiber was significantly influenced by the cellulose crystal size. The crystal size (CS) of the fiber was determined by the given below Scherer's empirical equation.

$$CS = \frac{(K\lambda)}{(\beta \cos \theta)} \quad (6)$$

Where,

$K = 0.89$  is Scherer's constant,

$\beta$  = Peak's full width at half-maximum,

$\lambda$  = The wavelength of radiation.

#### 4.8 SINGLE FIBER TENSILE STRENGTH

The tensile resistance of natural fibers was assessed through a meticulous single fiber tensile test, employing a sample size and standards compliant with ASTM 3822–07. In this evaluation, thirty randomly chosen fiber samples are tested each of gauge length 40mm.

Zwick/Roell Universal testing machine with a 5kN capacity load cell was used for the tensile testing of the fiber. This versatile equipment allows for different load cell and crosshead speed settings, accommodating the diverse characteristics of natural fibers. In this instance, 5 mm/min crosshead speed was maintained during the test, ensuring a controlled and precise assessment of the fiber's tensile strength.

#### 4.9 THERMOGRAVIMETRIC ANALYSIS (TGA)

The thermal stability of the *Grewia villosa* fiber was determined by the thermogravimetric analysis. To carry out this analysis, a sophisticated instrument known as the Jupiter simultaneous thermal analyzer was employed. The experimental procedure began by subjecting a powdered sample of the natural fiber to controlled heating. The temperature gradually increased from room temperature to an impressive 1000°C, with a consistent heating rate of 10°C per minute. Notably, this entire process occurred in a nitrogen atmosphere. This choice of atmosphere was deliberate, aimed at preventing any unwanted oxidation effects and ensuring the reliability of the results. Continuous supply of nitrogen which was highly pure was supplied to the furnace at 20 mL per minute flow rate throughout the analysis.

#### 4.10 SCANNING ELECTRON MICROSCOPY (SEM)

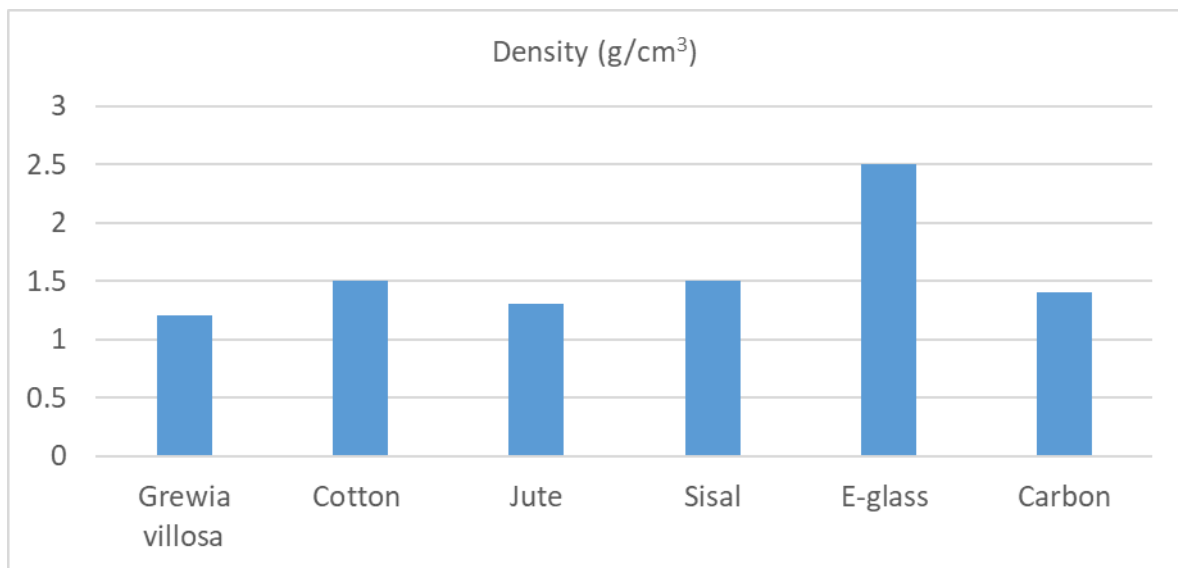
The *Grewia villosa* fiber external surface was examined through scanning electron microscope. In this analysis, the fiber samples underwent a meticulous preparation process. Initially, they were affixed onto carbon adhesive tape, securely attached to a brass disc. This ensured the stable positioning of the samples during imaging. The SEM imaging was performed using a high-energy electron beam with an accelerating voltage of 10 kV. To create the necessary vacuum environment for precise imaging, a pressure of  $1.5 \times 10^{-3}$  Pa was maintained throughout the experiment. Before commencing the study, a thin layer of gold, approximately 10 nm thick, was meticulously deposited onto the fiber samples. This gold layer was applied using a 15 kV sputter coater. This preparatory step was essential to prevent charge accumulation during the imaging process, ensuring accurate and detailed examination of the fiber's surface morphology.

## CHAPTER 5

### RESULTS AND DISCUSSION

#### 5.1 PHYSICAL ANALYSIS

The physical analysis of natural fibers plays a pivotal role in characterization of fiber. The density of the fiber indicates the weight of the fibre. The calculated density of Grewia Villosa fiber was  $1.21 \text{ g/cm}^3$ . The density of the Grewia villosa fiber was lower than the synthetic fibers like Glass (E) ( $2.5 \text{ g/cm}^3$ ), carbon ( $1.4 \text{ g/cm}^3$ ), and boron ( $2.8 \text{ g/cm}^3$ ). The traditional fibers like jute ( $1.45 \text{ g/cm}^3$ ), hemp ( $1.47 \text{ g/cm}^3$ ) and sisal ( $1.5 \text{ g/cm}^3$ ) also have high density as compared to Grewia villosa fiber. Due to the less density, the fiber can be utilized for light weight applications. The figure 5.1 shows the comparison of GVF density with other bark fibers.



**Fig 5.1: Density Comparison Graph**

The determination of diameter of the fiber was difficult due to the uneven profiles. However, the fiber shape was accepted to be circular, and the diameter was measured at three different instances and its average was taken as the diameter value. The measured diameter of the Grewia villosa was  $144 \pm 0.11 \text{ }\mu\text{m}$ .

## 5.2 CHEMICAL ANALYSIS

Chemical analysis of natural fibers is a crucial process that helps in understanding their composition and properties. The natural fibers importantly comprise of cellulose, lignin, hemicellulose, wax, moisture content and ash. Various techniques are applied to determine the chemical composition of natural fibers. The cellulose content of the *Grewia villosa* fiber (65.16 wt. %) was quite high. Cellulose is of paramount importance in natural fibers due to its unique properties and widespread presence in plant cell walls. As the primary structural component of plant tissues, cellulose provides natural fibers with exceptional strength and durability. Table 5.1 reveals the chemical analysis of various bark fibers.

*Table 5.1: Chemical Analysis of Bark Fibers*

| Fiber                        | Cellulose (wt%) | Hemicellulose (wt%) | Lignin (wt%) | Ash (wt %) | Moisture content (wt%) | Wax (wt %) | Reference                                        |
|------------------------------|-----------------|---------------------|--------------|------------|------------------------|------------|--------------------------------------------------|
| <i>Grewia villosa</i>        | 65.16           | 31.5                | 15.02        | 5.9<br>2   | 8.24                   | 0.54       | Current study                                    |
| <i>Acacia leucophloea</i>    | 68.09           | 13.6                | 17.73        | 0.0<br>8   | 8.83                   | 0.55       | (Arthanarieswaran et al., 2015)                  |
| <i>Acacia planifrons</i>     | 73.1            | 9.41                | 12.04        | 4.0<br>6   | 8.21                   | 0.57       | (Senthamaraikannan, Saravanakumar, et al., 2019) |
| <i>Albizia amara</i>         | 64.54           | 14.32               | 15.61        | 4.1        | 9.34                   | 0.56       | (Senthamaraikannan, Sanjay, et al., 2019)        |
| <i>Albizia Saman</i>         | 60.76           | 16.97               | 14.64        | 6.8<br>3   | 11.71                  | 1.5        | (Gopinath et al., 2022)                          |
| <i>Carica papaya</i>         | 58.71           | 11.8                | 14.26        | 4.7        | 9.73                   | 0.81       | (Saravana Kumaar et al., 2019)                   |
| <i>Ceiba pentandra</i>       | 60.9            | 17.53               | 23.5         | 1.0<br>5   | 7.46                   | 0.38       | (Kumar et al., 2018)                             |
| <i>Dichrostachys Cinerea</i> | 72.4            | 13.08               | 16.89        | 3.9<br>7   | 9.82                   | 0.57       | (Baskaran et al., 2018)                          |
| <i>Ficus Carica</i>          | 63.17           | 12.08               | 13.7         | 1.0<br>5   | 9.07                   | 0.42       | (Manivel Selvaraj et al., 2023)                  |
| <i>Ficus Racemosa</i>        | 72.36           | 11.21               | 10.45        | 6.5<br>9   | 6.13                   | 0.25       | (Manimaran, Saravanan, et al., 2021)             |
| <i>Prosopis juliflora</i>    | 61.65           | 16.14               | 17.11        | 5.2        | 9.48                   | 0.61       | (Saravanakumar et al., 2013)                     |
| <i>Thespesia populnea</i>    | 70.27           | 12.64               | 16.34        | 1.8        | 10.83                  | 0.76       | (Kathirselvam et al., 2019)                      |
| <i>Tithonia Diversifolia</i> | 63.9            | 19.4                | 19.3         | 3.4        | 9.8                    | 0.61       | (Gopinath et al., 2023)                          |
| <i>Vachellia farnesiana</i>  | 38.3            | 12.1                | 9.2          | 6.2<br>1   | 11                     | 3.4        | (Vijay et al., 2022)                             |
| <i>Albizia Lebbeck</i>       | 72.59           | 9.69                | 10.08        | 4.2<br>3   | 8.56                   | 0.52       | (Manimaran, Solai Senthil Kumar, et al., 2021)   |

Hemicellulose is a branched polysaccharide that makes up a significant portion of plant cell walls, alongside cellulose and lignin. One of the key functions of hemicellulose in natural fibers is its ability to bind cellulose microfibrils together. This bonding provides structural integrity to plant cell walls, enhancing the overall strength and flexibility of natural fibers. Hemicellulose also acts as a glue-like substance that helps hold cellulose fibers together, making them less susceptible to breaking or splitting. Additionally, hemicellulose has the ability to absorb and release moisture efficiently. The hemicellulose content of the fiber was known to be 31.5 wt. %.

Lignin is a complex organic polymer found in the cell walls of various natural fibers, playing a crucial role in providing structural support and rigidity to plants. *Grewia villosa* possesses 15.02 wt. %. This stops the entry of enzymes which harms the fiber through cell walls and improves the mechanical strength of fiber.

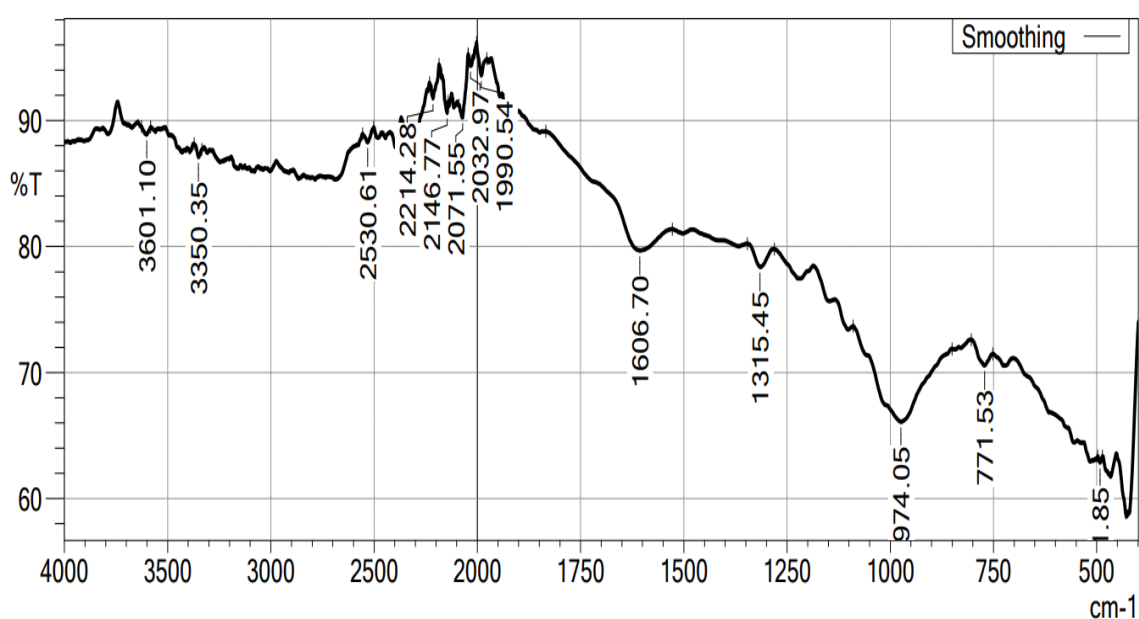
Wax is a natural component found in various plant fibers, contributing to their resilience and durability. The wax decreases the strength between the fiber and the matrix bond. Positively, the *Grewia villosa* possesses 0.54 wt. % which may indicate the better bonding between the fiber and resin.

Ash content has a vital role in the context of natural fibers which acts as a reinforcement in composite materials. This ash content can impact the mechanical properties and fire resistance of the composite. High ash content can lead to increased stiffness but may also make the composite more brittle. Engineers and manufacturers carefully consider ash content when designing composite materials, as it can influence the final product's strength, durability, and suitability for specific applications in industries like automotive, construction, and aerospace. The *Grewia villosa* has low ash content of 5.92 wt. %. This makes the fiber more flexible.

Moisture content is a significant factor which has impact on the composite's mechanical properties and overall performance. High moisture content can weaken the fiber-matrix bond, reduce composite strength, and potentially lead to mold or fungal growth. The fiber has 8.25 wt.% of moisture content.

### 5.3 FTIR ANALYSIS

FTIR is used to determine major chemical functional groups in the GVF. A peak at  $3350.35\text{ cm}^{-1}$  responsible for O-H stretching due to the presence of  $\alpha$ -cellulose.  $2214.28\text{ cm}^{-1}$  peak symbolizes the C=C stretching of wax. A broad peak at  $1606.7\text{ cm}^{-1}$  represents C=O stretching vibration of the  $\alpha$ -keto carboxylic acid in lignin. A peak at  $1315.45\text{ cm}^{-1}$  was due to C=O stretching of hemicellulose. A deep and wider peak at  $974.05\text{ cm}^{-1}$  was due to C-OH stretching vibration of lignin. The figure 5.2 shows the FTIR graph of GVF.

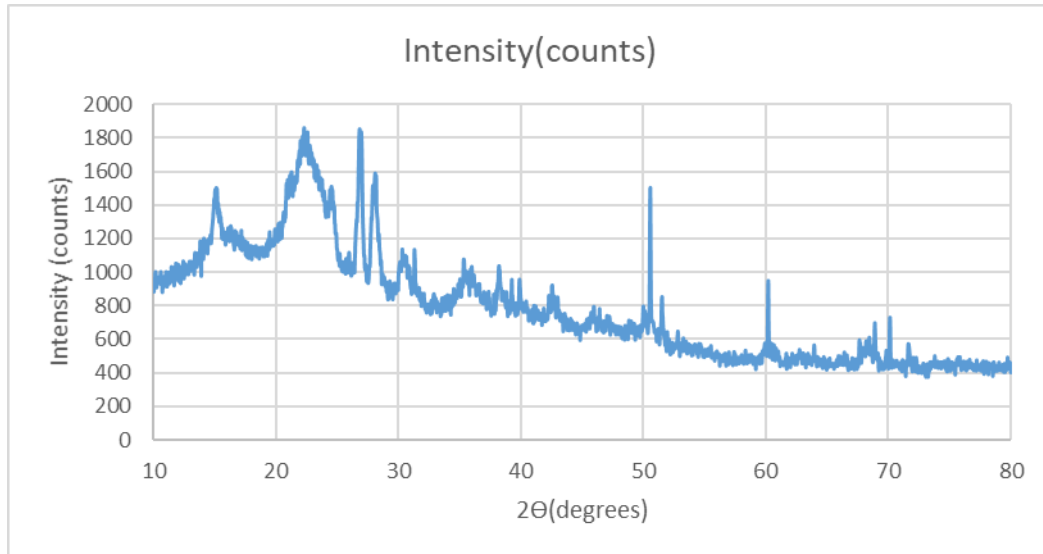


**Fig 5.2: FTIR Graph of GVF**

### 5.4 XRD ANALYSIS

Figure 5.3 indicates the XRD pattern of the *Grewia Villosa*. From the figure it is known that the two main peaks at the peak at  $2\theta = 15.15^\circ$  correlates to the crystallographic plane (1 1 0), showing amorphous material (amorphous cellulose, pectin, lignin, and hemicelluloses) in the GVF's. The peak at  $2\theta = 22.45^\circ$  is the crystallographic plane (2 0 0), which drives crystalline material. The crystalline value of the GVF was determined as 48.22% which is comparatively greater than *Acacia Concinna* (27.5%), *Ficus Carica* (41.42%), *Silybum marianum* (45%), and *Cissus vitiginea* (30.5%). The crystalline size (CS) of the fiber was determined as 26.97 nm

which is higher than *Ceiba pentandra* (22.48 nm), *Albizia Lebbeck* (16.57 nm) and *Cissus vitiginea* (12.69 nm).



**Fig 5.3: XRD Graph of GVF**

## 5.5 SINGLE FIBER TENSILE TEST

Single fiber tensile testing is another vital method to evaluate a fiber's mechanical properties. This test assesses factors like tensile strength, modulus, and elongation at break, providing valuable information about the fiber's strength. The tensile strength of the *Grewia villosa* fiber was determined and it was  $160.74 \pm 107.02$  MPa with a strain rate of  $2.93 \pm 1.31\%$ . The GVF's young's modulus was calculated as  $6.02 \pm 3.89$  MPa.

The micro fibrillar angle ( $\alpha$ ) is a key factor in determining the mechanical durability of natural fibers. It indicates the angle at which cellulose microfibrils develop in relation to the fiber axis. Elevated microfibrillar angle of fiber demonstrate excellent ductility, whereas those with a small microfibrillar angle have high strength. The microfibrillar angle (MFA) of *Grewia Villosa* was estimated using the deformation equation and found to be  $1.03^\circ$ - $13.36^\circ$ . Natural fibers with high ductility, such as Coir, Cotton, and Sisal, with MFA values of  $44^\circ$ ,  $25^\circ$ , and  $16^\circ$ , and failure strains of 27.5%, 7%, and 8%, respectively.

$$\varepsilon = \ln\left(1 + \frac{\Delta L}{L_0}\right) = -\ln \cos \alpha \quad (7)$$

Where,

$\varepsilon$  is the strain at failure

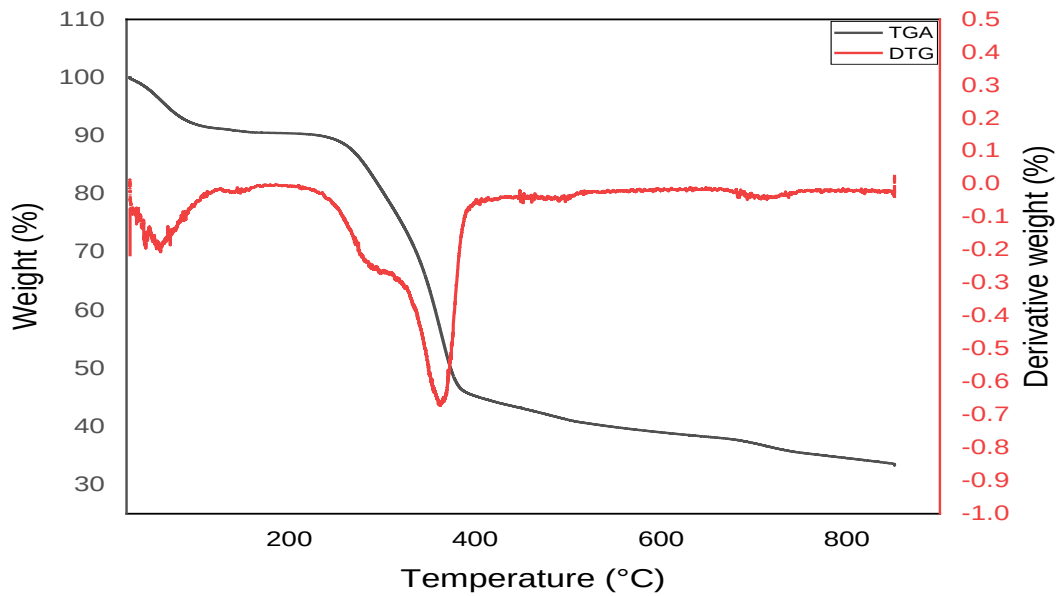
$\Delta L$  is the elongation at break in mm

$L_0$  is the gauge length in mm, and

$\alpha$  is the micro fibrillar angle (deg).

## 5.6 THERMOGRAVIMETRIC ANALYSIS (TGA)

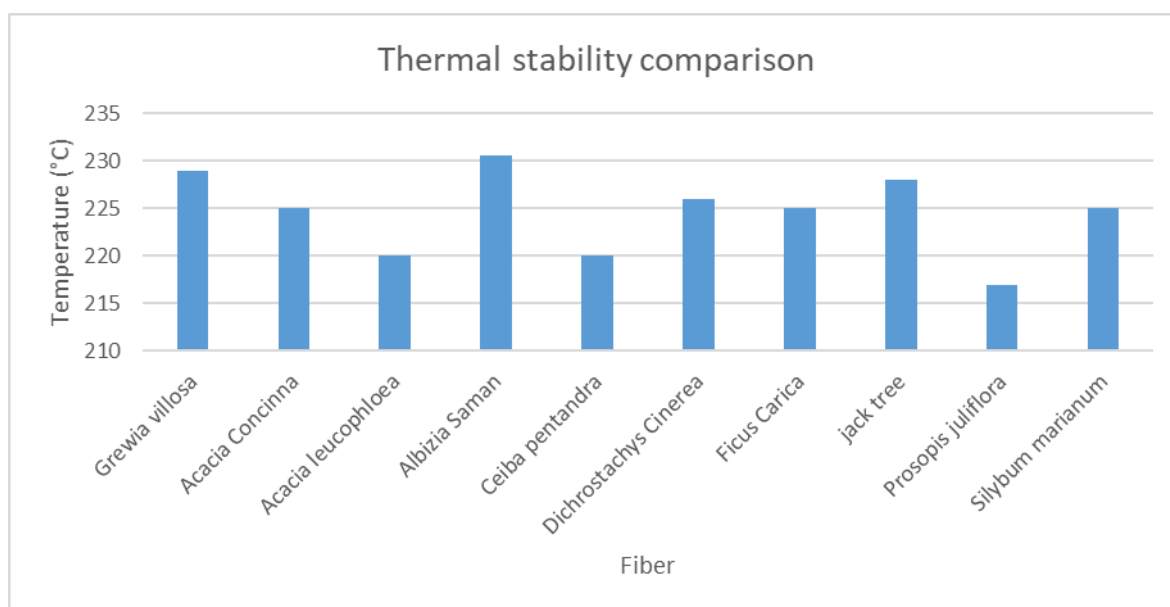
TGA and DTG curves which are shown in figure 5.4 were used to determine the thermal behavior of the GVF.



**Fig 5.4: TGA and DTG Graph of GVF**

The initial degradation was observed at 60°C primarily due to the evaporation of water content with mass loss of 9.97%. The thermal stability of GVF was 219.32°C. The secondary peak was observed at 296°C represents the 43.55% mass loss due to depolymerization of hemicellulose. Another major peak observed at 363.49°C

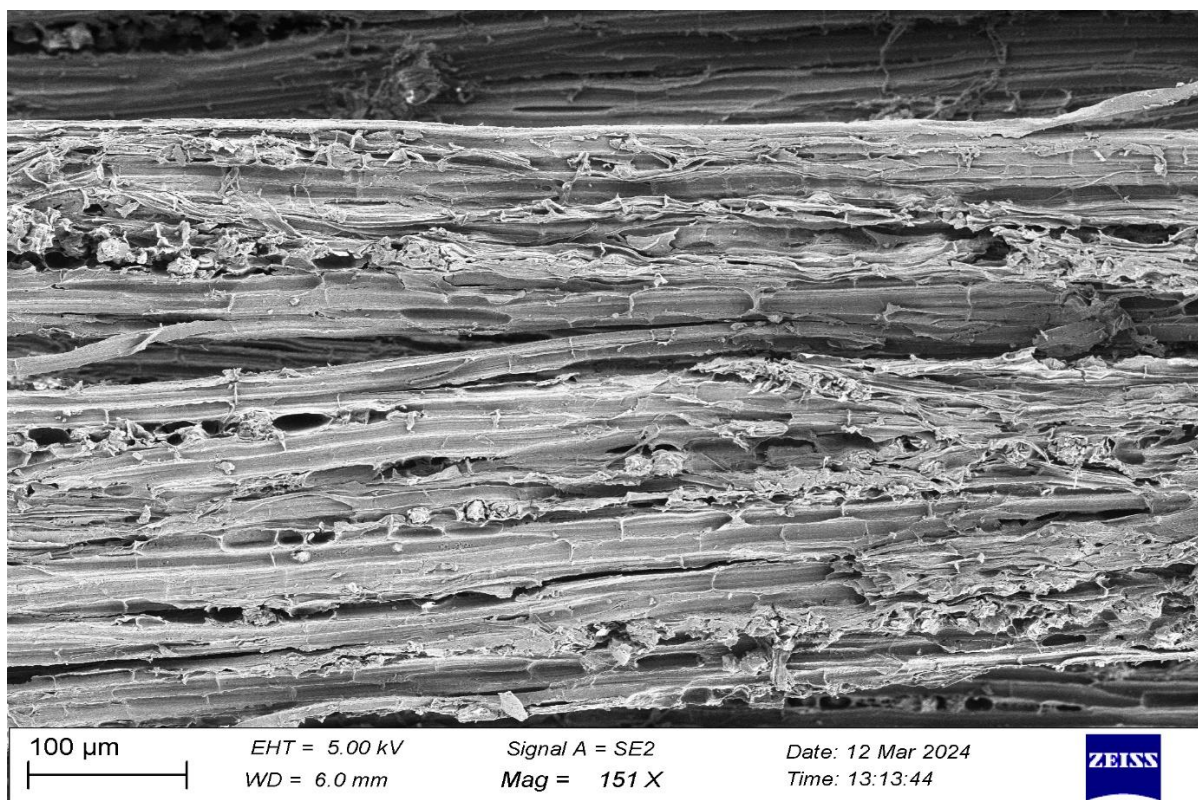
signifies the lignocellulose degradation with mass loss of 13.21%. Similar peaks observed for Acacia Caesia (308°C), Acacia Concinna (326°C), Albizia amara (330.6°C), Ficus Racemosa (310.5°C), Thespesia populnea (323.76°C) and Dichrostachys Cinerea (359.3°C). Figure 5.5 shows the comparison of thermal stability of GVF with other bark fibers.



**Fig 5.5: Thermal Stability Comparison**

## 5.7 MORPHOLOGICAL ANALYSIS

Field Emission Scanning Electron Microscopy (FESEM) with high resolution was utilized for the morphological study of GVF. The figure 5.6 displays the morphology of the GVF. The white colored micro particles represent the lignin and hemicellulose which are amorphous in nature present in the fiber. Impurities were also observed in the morphological study. It is a bundle of microfibrils aligned in an axis. The GVF's surface is rough and because of that it can bond strongly with the matrix. The pores were not observed in the fiber surface. It represents the higher strength of the fiber. Due to the absence of the pores the fiber can't be used for acoustic applications.



**Fig 5.6: SEM Image of GVF**

## CHAPTER 6

### SUMMARY AND CONCLUSION

#### 6.1 SUMMARY

The *Grewia villosa* tree bark fiber was collected and fiber was extracted from the bark. The fiber was dried in oven to get rid of moisture. The fiber was undergone different types of characterization tests which includes physical analysis, chemical analysis, single fiber tensile strength, FTIR, XRD, TGA and SEM. The density of the fiber was  $1.21 \text{ g/cm}^3$  which was lower than synthetic and renowned natural fibers. The GVF has quite high cellulose content of 65.16%. The fiber achieved an average tensile strength of  $160.74 \pm 107.02 \text{ MPa}$  with strain rate of  $2.93 \pm 1.31\%$ . The young's modulus of the fiber was  $6.02 \pm 3.89 \text{ GPa}$ . The fiber achieved a thermal stability of  $219.32^\circ\text{C}$  which is high compared to *Acacia Concinna*, *Albizia saman* etc. The fiber has crystallinity of 49% and crystal size of 27.28 nm. The high crystal size minimizes the surface area of the fiber thereby decreasing the reactivity of the fiber with other chemicals and the water vapour.

#### 6.2 CONCLUSION

The fiber extraction and characterization of was done. The GVF's density was very low so that the fiber can be used for low weight applications. The fiber has high cellulose percentage which contributes to the mechanical strength of it. The GVF's thermal stability was also very high so that it can be used for high temperature applications for below  $200^\circ\text{C}$ . The crystallinity of the fiber was good which will enhance the mechanical strength of GVF. The crystal size of the fiber was high which reduces the surface area accessible to the environment there by reducing the reactivity with chemicals and vapour. The SEM images indicates no porosity and rough surface. Overall, the fiber was good substitute for man-made fibers to use in reinforcement of composites of low strength and high thermal stability.

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## APPENDIX



***Leaves and Fruits of Grewia Villosa Tree***



***Stripped Bark from Grewia Villosa***



***Grewia Villosa Flower***