DEVELOPMENT AND MORPHOLOGICAL ANALYSIS OF SUSTAINABLE BIO-HYBRID COMPOSITES FOR BRAKE PAD APPLICATION

Submitted by

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ABSTRACT

Biocompatible automobile materials have received attention due to their potential to reduce environmental concerns. For automotive hybrid composites, this study evaluates and monitors the interfacial and mechanical properties of an epoxy glue with natural fillers, Loofah shell and teak wood powder. This study examines how natural fillers synergistically improve epoxy adhesive's mechanical strength, biocompatibility, and interfacial adhesion, essential for automotive applications. In-situ monitoring simulates the physiological milieu in automotive applications by measuring mechanical behavior and interfacial adhesion in real time under various environmental circumstances. This study found that the optimized formulation has good mechanical properties and biocompatibility, making it suitable for brake pads, clutch facings, and other non-structural automotive components that require friction, wear resistance, and environmental sustainability. This research promotes sustainable and biocompatible

automotive engineering materials, delivering new lightweight component production and ecologically friendly materials.

CHAPTER 1

INTRODUCTION

In recent years, the growing demand for sustainable and eco-friendly materials has led to the development of natural fiber-reinforced polymer composites. Among these, hybrid composites that combine two or more natural reinforcements are gaining attention for their enhanced mechanical and environmental properties. One such promising combination is loofah fiber and teak wood powder used in hybrid composite materials. Loofah fiber, derived from the matured fruit of the *Luffa cylindrica* plant, is a lightweight, biodegradable material known for its high porosity and good mechanical strength. It has a cellulose-rich structure that offers decent reinforcement when used in polymer matrices.

Teak wood powder, a byproduct of teak wood processing, is a finely ground, lignocellulosic material. It contributes to the mechanical strength and thermal stability of composites while offering a way to recycle waste material from the timber industry. By combining loofah fiber's structural reinforcement with the filler effect and thermal resistance of teak wood powder, hybrid composites can achieve a balance of mechanical strength, biodegradability, and cost-effectiveness. These materials are suitable for applications in automotive interiors, construction panels, furniture components, and packaging, especially where environmental considerations are important. The development of loofah fiber and teak wood powder hybrid composites aligns with the global shift toward green materials and circular economy principles, offering a sustainable alternative to synthetic fiber composites.

The automotive industry is shifting toward sustainable materials, and bio-hybrid composites offer an eco-friendly alternative for brake pads. Traditional brake pads release pollutants and are non-biodegradable, whereas bio-hybrid composites, made from natural fibers and polymers, reduce environmental impact. These materials improve fuel

efficiency, reduce weight, and comply with strict regulations. Morphological analysis ensures their durability, wear resistance, and thermal stability. In this context, the incorporation of natural fillers such as loofah, coconut coir and jute fiber into epoxy adhesives presents an intriguing avenue for the development of bio-hybrid composites tailored for biomedical purposes. These natural fillers, abundant and renewable resources, offer unique advantages including biocompatibility, low cost, and eco-friendliness.

Moreover, their inherent properties such as high strength, lightweight nature, and biodegradability make them attractive candidates for enhancing the mechanical and interfacial properties of epoxy adhesives. The evaluation and in-situ monitoring of the interfacial and mechanical properties of epoxy adhesive-based bio-hybrid composites hold significant importance in ensuring their efficacy and suitability for biomedical applications. Understanding the synergistic effects of natural fillers on the mechanical strength, adhesion, and biocompatibility of epoxy composites is crucial for the design and optimization of materials tailored to specific automotive requirements.

Figure 1.1 Loofah Shell Fiber

1.2 Loofah Fiber

Figure 1.3 Wood Powder

This research aims to address this need by systematically investigating the interfacial and mechanical properties of epoxy adhesive- based bio-hybrid composites incorporating pistachio powder, eggshell, and rice husk. Advanced characterization techniques will be employed to analyze the microstructural features, chemical interactions, and surface morphology of the developed composites. Additionally, mechanical testing protocols and in-situ monitoring techniques will be utilized to assess the real-time performance and behavior of these composites under simulated physiological conditions. By elucidating the interplay between natural fillers and epoxy adhesives, this research endeavors to contribute to the development of sustainable and biocompatible materials with enhanced mechanical properties for biomedical engineering applications. Ultimately, the outcomes of this study have the potential to pave the way for the advancement of innovative solutions in healthcare, ranging from tissue engineering scaffolds to medical implants, while promoting environmental sustainability. Figure 1.1, 1.2 and 1.3 shows the loofah and wood powder.

1.2COMPOSITE MATERIALS

Bio-hybrid composite materials refer to a class of materials that incorporate both synthetic and natural components, designed for various biomedical applications. These materials typically combine synthetic polymers, such as epoxy resins, with natural fillers or reinforcements derived from biological sources, such as plant fibers, cellulose, chitosan, or agricultural by-products. The integration of natural components into synthetic matrices aims to harness the desirable properties of both constituents, resulting in composites that exhibit enhanced mechanical strength, biocompatibility, and sustainability.

Bio-hybrid composites offer several advantages over traditional synthetic materials in biomedical applications. Firstly, the incorporation of natural fillers can improve the biocompatibility of the composite, reducing the risk of adverse reactions when used in contact with biological tissues or fluids. Secondly, natural fillers often contribute to the lightweight nature of the composite, making them suitable for applications where weight considerations are crucial, such as implants or wearable devices. Additionally, the renewable and sustainable nature of natural fillers aligns with the growing emphasis on environmentally friendly materials in biomedical engineering.

These materials find diverse applications in biomedical engineering, including tissue engineering scaffolds, drug delivery systems, medical implants, wound dressings, and diagnostic tools. The versatility of bio- hybrid composites allows for tailored designs to meet specific requirements of each application, such as mechanical strength, biodegradability, or bioactivity. Overall, bio-hybrid composite materials represent an innovative approach in biomaterials science, offering a balance between synthetic performance and natural sustainability for a wide range of biomedical applications Figure 1.4 shows various Bio Hybrid Composites.

Figure 1.4 Bio Hybrid Composites

1.2.1TYPES OF COMPOSITE MATERIAL

Composite materials are engineered by combining two or more distinct materials to achieve superior properties that the individual components alone cannot provide. These components are generally classified as the matrixand the reinforcement**.** The matrix binds the reinforcement together, while the reinforcement provides strength and stiffness to the composite. Composite materials can be classified into several types based on the matrix material**,** reinforcement type**,** andstructure**.** Below is a detailed classification:

1. Classification Based on Matrix Material

A. Polymer Matrix Composites (PMCs)

- **Matrix:** Thermosetting or thermoplastic polymers
- **Reinforcement:** Fibers like glass, carbon, aramid, or natural fibers **Advantages:** Lightweight, corrosion-resistant, easy to process
- **Applications:** Aerospace, automotive parts, sporting goods, marine structures
- **Examples:**
  - Glass Fiber Reinforced Polymer (GFRP)
  - Carbon Fiber Reinforced Polymer (CFRP)
  - Natural fiber composites (e.g., jute, hemp, loofah-reinforced polymers)

B. Metal Matrix Composites (MMCs)

- **Matrix:** Metals like aluminum, titanium, or magnesium
- **Reinforcement:** Ceramic fibers or particles (e.g., silicon carbide, boron carbide)
- **Advantages:** Higher strength, stiffness, and thermal resistance than PMCs
- **Applications:** Aerospace, automotive, electronics, military armor

Examples:

- Aluminum reinforced with silicon carbide particles
- Titanium matrix with carbon fibers

C. Ceramic Matrix Composites (CMCs)

- **Matrix:** Ceramic materials like alumina, silicon carbide
- **Reinforcement:** Ceramic fibers, whiskers
- **Advantages:** High-temperature stability, corrosion resistance, wear resistance
- **Applications:** Gas turbines, brake disks, aerospace heat shields
- **Examples:**
  - SiC/SiC composites
  - Al_2O_3 reinforced with zirconia fibers

D. Carbon-Carbon Composites

- **Matrix and reinforcement:** Both are carbon-based
- **Advantages:** Extremely high-temperature resistance, excellent strength-to-weight ratio
- **Applications:** Rocket nozzles, aircraft brakes, space structures

2. Classification Based on Reinforcement Form

A. Fiber-Reinforced Composites

- **Short Fiber-Reinforced Composites:** Fibers <1 mm, randomly oriented
- **Long Fiber-Reinforced Composites:** Fibers >1 mm, aligned or woven
- **Continuous Fiber Composites:** Uninterrupted fibers throughout the matrix
- **Discontinuous Fiber Composites:** Chopped or randomly distributed fibers

B. Particulate-Reinforced Composites

- **Reinforcement:** Small particles (metal, ceramic, or polymer)
- **Shape:** Spherical, irregular, or flake-like
- **Examples:** Concrete (cement + sand), metal matrix composites with ceramic particles

C. Structural Composites

□**Laminate Composites:** Layers of different materials bonded together □**Sandwich Composites:** Two strong outer skins with a lightweight core □**Applications:** Aircraft wings, marine panels, building materials **3. Classification Based on Structure**

A. Laminar Composites

□**Made of:** Two or more layers bonded together

□**Features:** Different orientations enhance strength and stiffness

□**Examples:** Plywood, fiber-reinforced plastic laminates

B. Sandwich Composites

□**Structure:** Strong outer face sheets and lightweight core (e.g., foam or honeycomb)

□**Applications:** Aerospace, automotive, packaging C. Reinforced Composites

□**Structure:** Reinforcements embedded in a matrix in a systematic pattern

□**Used for:** Load-bearing applications

4. Classification Based on Origin of Reinforcement

A. Synthetic Fiber Composites

□**Reinforcements:** Glass fibers, carbon fibers, aramid (Kevlar)

□**Advantages:** High strength, predictable performance

B. Natural Fiber Composites (Bio-Composites)

□**Reinforcements:** Jute, flax, hemp, loofah, coir, sisal

□**Matrix:** Usually biodegradable or thermoplastic polymers □

Advantages: Renewable, biodegradable, eco-friendly, low cost

□**Applications:** Automotive panels, packaging, household items

Table 1.1: Comparison Table of Composite Materials

Type	Matrix	Reinforcement	Key Properties	Applications
PMCs	Polymers	Fibers (glass, carbon, natural)	Lightweight, corrosion-resistant	Aerospace, automotive
MMCs	Metals	Ceramics, fibers	High strength, thermal resistance	Aerospace, engine parts
CMCs	Ceramics	Ceramic fibers	High temp resistance	Turbines, brakes
Bio-Composites	Biopolymer	Natural fibers	Eco-friendly, biodegradable	Packaging, automotive interiors

1.3 EVOLUTION OF COMPOSITES

Composites are engineered materials made by combining two or more constituent materials with significantly different physical or chemical properties. The evolution of composites has occurred over thousands of years and can be categorized into several key stages:

1. Ancient Composites (Prehistoric – ~1000 AD)

- **Examples:** Mud bricks reinforced with straw, plywood in Egypt (1500 BC), and laminated bows.
- **Materials Used:** Natural fibers (straw, wood), resin-like substances (tree sap), bitumen.
- **Applications:** Housing (mud bricks), weaponry (bows), and water containers.

Straw-reinforced mud bricks used in ancient Mesopotamia were among the first examples of composites.

2. Early Engineered Composites (1000 – 1800s)

- **Examples:** Laminated wood (plywood), reinforced boats using tar and animal hair.

- **Materials Used:** Wood veneers, natural adhesives.
- **Applications:** Building construction, ships, furniture.

In China and Greece, layers of wood were glued together in alternating grain directions to improve strength.

3. Industrial Revolution (1800s – 1930s)

- **Key Developments:** Increased use of metals and discovery of polymers.
- **Materials Used:** Metal matrix with natural fiber, early thermosetting resins.
- **Applications:** Machinery, building materials.

Early thermosetting plastics like Bakelite (1907) opened new doors for engineered materials.

4. Birth of Modern Composites (1940s – 1960s)

- **Key Milestone:** Development of **glass fiber** in the 1930s and **polyester resin** in the 1940s.
- **Result:Glass fiber reinforced polymer (GFRP)** composites.
- **Applications:** Aerospace (WWII aircraft), automotive, marine industries.

Glass fiber revolutionized composite material use due to high strength-to-weight ratio.

5. Advanced Composites Era (1970s – 1990s)

- **Key Innovations:**
  - **Carbon fiber** and **aramid fiber (Kevlar)**.
  - Use of **epoxy resins**.
- **Applications:** Aerospace (Boeing, military jets), sports equipment, racing cars.

These materials provided exceptional strength, stiffness, and temperature resistance.

6. Nanocomposites and Bio-Composites (2000s – Present)

- **Nanocomposites:** Incorporation of nano-fillers (e.g., carbon nanotubes, nanoclays).
- **Bio-composites:** Use of **natural fibers (jute, hemp, flax, loofah)** and **biodegradable polymers**.
- **Applications:** Medical, environmental, automotive, construction.

7. Smart and Multifunctional Composites (2010s – Future)

- **Smart composites:** Materials that respond to stimuli (temperature, electric field).
- **Self-healing**, **self-sensing**, and **energy-harvesting** composites.
- **Applications:** Robotics, aerospace, wearable electronics.

The evolution of composites showcases humanity's continuous pursuit of stronger, lighter, and more sustainable materials. From mud bricks to smart nano-bio-hybrids, composites have evolved with technological demands — culminating in today's use of **natural fibers** like **loofah** in eco-friendly applications.

Table1.2: Evolution of Composites

Era / Period	Time Frame	Key Materials	Technological Milestones	Applications
Ancient Composites	Prehistoric – 1000 AD	Straw + mud, wood + resin, tree sap, bitumen	First man-made composites like mud bricks and laminated wood	Housing, water containers, weapons (bows)
Early Engineered	1000 – 1800s	Wood veneers, natural glue, hair reinforcement	Plywood and natural laminates used in boats and furniture	Construction, shipbuilding, furnishings
Industrial Revolution	1800s – 1930s	Metal matrices, early polymers (e.g., shellac), rubber	Mass production, early polymer development	Building, machines, early transportation
Modern Composites	1940s – 1960s	Glass fiber, polyester resin, early thermosets	Birth of GFRP (Glass Fiber Reinforced Plastic)	Aircraft, boats, automotive
Advanced Composites	1970s – 1990s	Carbon fiber, Kevlar, epoxy resins	High-performance composites with high strength-to-weight ratios	Aerospace, defense, sports, racing
Bio & Nanocomposites	2000s – Present	Natural fibers (jute, hemp, loofah), nano-fillers	Rise of eco-friendly, biodegradable, and nano-engineered materials	Green buildings, automotive panels, biodegradable goods
Smart Composites	2010s – Future	CNTs, graphene, self-healing polymers, piezoelectric fibers	Self-sensing, self-healing, and multifunctional composites	Wearables, aerospace, robotics, structural monitoring

1.3HYBRID COMPOSITE MANUFACTURING PROCESSES

Hybrid composites are materials made by combiningtwo or more types of fibers or reinforcements within a single matrix system. The manufacturing methods aim to ensure uniform dispersion, good bonding**,** and desired orientation of the reinforcing materials.

1. Hand Lay-Up Process

Principle:

A manualtechnique where reinforcing fibers (woven or chopped) are placed in a mold and impregnated with liquid resin using brushes or rollers.

Steps:

1.Apply release agent on mold surface.

2.Place layers of natural (e.g., loofah) and synthetic fibers**.** 3.Pour or brush resin (e.g., epoxy) over each layer.

4.Use rollers to remove air bubbles and distribute resin. 5.Allow to cure at room temperature or with mild heating.

Equipment:

- Open mold
- Brushes, rollers
- Resin and curing agents
- Personal protective equipment

Advantages:

- Low-cost and simple
- Ideal for custom or experimental parts
- Good for natural fibers (jute, hemp, loofah)

Limitations:

- Labor-intensive
- Quality depends on operator skill
- Limited strength and consistency

2. Compression Molding

Principle:

A pre-measured fiber-resin mixture (called charge) is placed into a heated mold and pressed under high pressure to form the desired shape.

Steps:

1.Place hybrid fiber mats or preforms in mold cavity. 2.Add resin or use pre-impregnated material.

3.Close mold and apply heat + pressure.

4.Cure under compression and demold the part.

Equipment:

- Hydraulic press
- Matched die mold
- Heating elements

Advantages:

- Good dimensional accuracy
- Suitable for medium to high-volume production
- Excellent for bio-hybrid brake pads

Limitations:

- High tooling cost
- Limited to moderate complexity geometries

3. Resin Transfer Molding (RTM)

Principle:

A closed mold process where dry fibers are placed in the mold and resin is injected to impregnate them under pressure.

Steps:

1.Arrange natural and synthetic fibers in mold cavity. 2.Seal the mold completely.

3.Inject resin under pressure or vacuum.

4.Allow to cure under closed conditions**.**

Equipment:

- Closed mold with ports
- Resin injection system
- Vacuum pump (optional)

Advantages:

- High-quality surface finish on both sides
- Controlled fiber-resin ratio
- Less waste and better repeatability

Limitations:

- Expensive mold setup
- Difficult to control for very porous natural fibers **4. Vacuum**

Bag Molding

Principle:

A variant of the hand lay-up process where vacuum pressure compresses the fiber-resin layup under a flexible plastic bag.

Steps:

1.Lay fibers and resin over mold.

2.Cover with peel ply, breather cloth, and vacuum bag film. 3.Seal the edges and apply vacuum.

4.Cure under pressure (with or without heat).

Equipment:

- Vacuum pump
- Bagging film and sealant
- Breather and release layers

Advantages:

□Reduces voids and improves quality □Better fiber-resin ratio than hand lay-up □Lightweight tooling

Limitations:

□Setup time is longer

□Vacuum integrity must be perfect

5. Pultrusion

Principle:

A continuous manufacturing process where fibers are pulled through a resin bath and then a heated die to form constant cross-section profiles.

Steps:

1.Continuous fibers (e.g., glass + loofah) are pulled through a resin bath**.** 2.Resin-saturated fibers enter a heated die**.**

3.Polymerization occurs inside the die.

4.Part is pulled and cut to length**.**

Equipment:

□Fiber creel

□Resin bath

□Heated forming die

□Pulling system

Advantages:

□High strength and uniformity

□Suitable for structural elements

Limitations:

□Only for uniform shapes (rods, beams)

□Loofah must be processed into continuous forms (which is hard)

6.Filament Winding

Principle:

Continuous fibers pre-impregnated with resin are wound under tension onto a rotating mandrel in precise patterns.

Steps:

1.Load fiber spools (glass, carbon, aramid, hybrid). 2.Pass through resin bath.

3.Wind over a rotating mandrel.

4.Cure the part (at room temp or in oven). 5.Remove the finished shell from mandrel.

Equipment:

- Filament winder
- Resin bath
- Rotating mandrel

Advantages:

- High fiber alignment
- Automated and repeatable
- Strong cylindrical parts

Limitations:

- Only for round/spherical parts
- Not suitable for chopped or irregular fibers like raw loofah **7. Injection Molding**

Principle:

Thermoplastic composites (with chopped hybrid fibers) are melted and injected into a closed mold to form complex shapes.

Steps:

1.Mix resin pellets and **short fibers** (natural + synthetic). 2.Feed into heated barrel.

3.Material is injected into the mold cavity.

4.Part cools and **ejects automatically**.

Equipment:

- Injection molding machine
- Mold tooling

Advantages:

- High-speed, automated
- Complex part geometries

Limitations:

- Only for **short fiber** composites
- Thermoplastics only

Table 1.3: Suitability for Loofah-Based Bio-Hybrids

Process	Loofah Suitability	Scale	Complexity
Hand Lay-Up	Excellent	Lab / Low Volume	Simple
Compression Molding	Excellent	Medium Volume	Moderate
Resin Transfer Molding	Moderate	Medium	High (equipment cost)
Vacuum Bag Molding	Good	Low to Medium	Moderate
Pultrusion	Low	High Volume	Limited shape
Filament Winding	Not Suitable	Medium to High	Only cylindrical
Injection Molding	Possible if chopped	High Volume	Complex (fiber prep)

Table1.4: Applications of Hybrid Composites Based on Process

Process	Common Applications
Hand Lay-Up	Prototypes, panels, bio-hybrid components (e.g., loofah pads)
Compression Molding	Brake pads, panels, automotive components
RTM	Aerospace parts, bicycle frames
Pultrusion	Beams, rods, building profiles
Filament Winding	Pressure vessels, tanks, pipes
Injection Molding	Casings, brackets, dashboards

Table1.5: Hybrid Composite Manufacturing Processes

Manufacturing Process	Principle	Suitable Matrix	Fiber Types	Advantages	Limitations
Hand Lay-Up	Manual placement of fibers and resin on mold	Thermosets (epoxy, polyester)	Natural + synthetic (jute + glass)	Low cost, simple, good for small batches	Labor intensive, low precision
Compression Molding	Pressing preform under heat and pressure	Thermosets, Thermoplastics	Chopped fibers, woven mats	High strength, good surface finish	High tooling cost, limited to specific shapes
Resin Transfer Molding (RTM)	Resin injected into a closed mold with dry fiber preform	Thermosets (epoxy, vinyl ester)	Complex hybrid fabrics	High quality, consistent, good for medium volumes	Requires accurate control of resin flow
Pultrusion	Continuous pulling of fibers through resin bath and die	Thermosets	Continuous fibers (glass, carbon)	Uniform shape, high production rate	Only for constant cross-sections
Filament Winding	Winding resin-impregnated fibers over rotating mandrel	Thermosets	Continuous hybrid yarns	High strength in hoop direction, automated	Limited to cylindrical/spherical shapes
Injection Molding	Molten matrix + chopped fibers injected into mold	Thermoplastics	Short natural + synthetic	High speed, suitable for small parts	Not ideal for continuous fiber structures

CHAPTER-2

LITERATURE REVIEW

2.1BIO-HYBRID COMPOSITES REINFORCED WITH NATURAL FIBERS

Jawaid et al. (2011) conducted a comprehensive study on the development of hybrid composites using jute and oil palm fibers as reinforcements in epoxy resin. Their research aimed to combine the mechanical strengths of jute with the cost-effectiveness and sustainability of oil palm fibers. The results showed significant improvements in tensile, flexural, and impact strengths for the hybrid composites compared to single-fiber-reinforced counterparts. The inclusion of both fibers also led to improved dimensional stability and moisture resistance. The study highlighted the potential of hybridizing natural fibers for enhancing composite properties while maintaining environmental benefits.

Sreekala and Thomas (2003) explored the use of oil palm fibers in unsaturated polyester resin, focusing particularly on the effects of surface treatments. Their study revealed that the mechanical properties, including tensile and flexural strength, were significantly enhanced when the fibers were treated chemically to improve adhesion with the matrix. This work emphasized the importance of fiber–matrix interfacial bonding in bio-composites and provided useful insights into tailoring composite properties through simple chemical modifications of natural fibers.

Chandramohan and Marimuthu (2011) fabricated and characterized hybrid natural fiber composites using sisal and coconut sheath fibers embedded in polyester resin. They focused on the damping, acoustic, and vibrational properties of the composites, which are essential for automotive and structural applications. Their findings showed that the inclusion of coconut sheath significantly improved damping capacity, while the sisal fiber contributed to tensile and flexural strength.

The study demonstrated the benefits of hybridization in achieving a balance of mechanical and functional properties.

Fiore et al. (2015) investigated the hybridization of flax and basalt fibers in an epoxy matrix to evaluate improvements in mechanical behavior. The combination of

natural (flax) and synthetic (basalt) fibers led to enhanced tensile, flexural, and impact properties due to the complementary mechanical characteristics of both fibers. Moreover, the hybrid composites exhibited superior interfacial bonding and improved energy absorption. This work supports the idea of using a bio-hybrid approach to achieve high-performance composites suitable for semi-structural applications.

Dhakal et al. (2007) studied the mechanical and water absorption behavior of jute–glass fiber-reinforced unsaturated polyester composites. The hybridization of jute with glass fibers led to notable improvements in tensile strength and impact resistance. Furthermore, the water absorption was reduced due to the presence of hydrophobic glass fibers. Their study provided a pathway to reduce the hydrophilic drawbacks of natural fibers by combining them with synthetic ones, making the composites more suitable for outdoor and semi-structural applications.

Ramesh et al. (2013) evaluated banana-jute-glass hybrid composites fabricated using hand lay-up technique. The study assessed the effect of fiber stacking sequence on tensile, flexural, and impact properties. It was found that a strategic placement of banana and jute layers between glass fiber layers provided enhanced load-bearing capacity and fracture resistance. The research highlighted the feasibility of using banana and jute fibers as eco-friendly alternatives to fully synthetic composites in moderate-load applications.

Jawaid et al. (2010) developed hybrid composites from kenaf and oil palm fibers in a polypropylene matrix and studied their tensile properties, void content, and chemical resistance. They found that the hybridization improved resistance to chemical degradation and reduced void formation due to better compaction and interfacial bonding. The study confirmed the viability of combining short and long natural fibers to balance strength and processing ease in thermoplastic matrices.

Sanjay and Yogesha (2016) provided an extensive review of the development of natural/glass fiber hybrid composites. They discussed how combining natural fibers like jute, flax, and coir with synthetic fibers enhanced the durability, stiffness, and strength of composites while retaining the biodegradability and cost advantages of natural fibers. The review highlighted the growing demand for such hybrids in the automotive and aerospace sectors due to their favorable strength-to-weight ratio and eco-friendliness.

Pickering et al. (2016) provided a critical review of the challenges and progress in using natural fibers in structural composites. Their research emphasized the effects of fiber alignment, matrix compatibility, moisture absorption, and treatments to improve fiber–matrix adhesion. The review concluded that hybridization and surface treatments were the most effective ways to enhance mechanical properties while ensuring biodegradability, thus making these composites viable for commercial use.

Noman et al. (2020) developed and analyzed bio-hybrid composites using hemp and basalt fibers in an epoxy matrix. The hybridization strategy yielded better tensile strength, thermal resistance, and flame retardancy compared to pure natural fiber composites. This work highlighted that carefully selected natural-synthetic combinations can offer both environmental benefits and mechanical reliability for high-performance applications like aerospace interiors.

John and Thomas (2008) explored the structural applications of hybrid composites made from banana and pineapple leaf fibers. Their study indicated that hybrid composites showed superior tensile and flexural behavior when compared to single-fiber counterparts. They also emphasized the potential of these fibers in replacing synthetic fibers in low to medium structural applications, especially in automotive interiors and furniture, due to their renewability, lightweight, and cost-effectiveness.

Suriani et al. (2018) fabricated hybrid composites using sugar palm and glass fibers in an unsaturated polyester matrix. Their mechanical testing revealed that sugar palm contributed to better energy absorption while glass fibers enhanced tensile and flexural strength. The study underlined the significance of fiber orientation and fiber type selection in achieving desirable hybrid performance, especially in semi-structural and automotive parts.

Saba et al. (2015) developed phenolic-based hybrid composites reinforced with kenaf and oil palm fillers. The hybrid composites exhibited improved hardness, tensile strength, and reduced water absorption compared to single-fiber composites. The researchers attributed this to the synergistic interaction between the fibers and the dense cross-linking of the phenolic resin. Their work supports the application of such hybrids in wet and thermally demanding environments.

Ahmed and Vijayarangan (2008) examined jute-glass fabric hybrid composites and tested their fatigue, flexural, and tensile properties. The results demonstrated significant gains in load-bearing capacity, fatigue life, and cost efficiency due to hybridization. This study showcased the economic and performance feasibility of using natural fibers with glass in structural applications such as marine and automotive panels.

Ku et al. (2011) reviewed a wide range of natural fiber composites and their mechanical behavior under different processing conditions. The study emphasized that chemical treatments such as alkali, silane, and peroxide significantly enhance the fiber–matrix interface. It also reported that hybridizing natural fibers with synthetic ones can offset the drawbacks of each and yield composites with balanced properties for engineering applications.

2.2 BIOBASED HYBRID COMPOSITE DESIGN FOR OPTIMUM HARDNESS AND WEAR RESISTANCE

George et al. (2016) investigated the use of banana and flax fibers reinforced with epoxy resin to enhance the wear resistance and surface hardness of biobased hybrid composites. Their study revealed that fiber hybridization
significantly improved both dry sliding wear performance and micro-hardness due to better stress distribution and interfacial bonding. The hybrid composite displayed less material loss under abrasive wear compared to single-fiber systems, making it suitable for moderate wear applications.

Saba et al. (2016) developed biobased phenolic composites using oil palm and kenaf fibers, focusing on wear resistance and hardness properties. Their tribological tests confirmed that the hybrid structure reduced wear loss and improved surface hardness significantly, attributed to the dense network formation in phenolic resin and the synergistic behavior of the two fibers. The research underlined the potential of such hybrids for automotive brake and clutch components.

Srinivasan et al. (2020) examined the mechanical and tribological behavior of natural hybrid composites reinforced with jute and hemp fibers in a polyester matrix. Their work demonstrated that the addition of hemp significantly improved wear resistance due to

its high cellulose content, while jute improved structural rigidity. The study recommended a 60:40 jute-hemp ratio for optimal hardness and wear performance.

Ali et al. (2019) conducted experiments on palm kernel shell (PKS) powder and basalt fiber hybridized in epoxy resin. Their study found that the biobased hybrid composite showed superior wear resistance under both dry and lubricated conditions, with a significant increase in surface hardness compared to composites with PKS alone. The hardness improvement was attributed to the fine particle size and interfacial adhesion.

Ramesh et al. (2014) focused on hybrid natural fiber composites using sisal and banana fibers with polyester resin. The addition of hard sisal fibers contributed to better wear resistance, while banana fibers helped balance toughness and impact resistance. The composite exhibited optimum hardness and lower specific wear rates under dry sliding conditions.

Nirmal et al. (2013) evaluated the dry sliding wear behavior of coconut sheath–reinforced polyester composites. Their findings showed that increasing the fiber content increased surface hardness and reduced wear loss. Hybridizing coconut sheath with glass fibers further enhanced these properties, making the composites suitable for sliding components in engineering applications.

Mishra and Biswas (2010) studied coir-jute hybrid composites with epoxy resin for wear resistance under varying loads. The results indicated that coir improved impact strength while jute provided wear resistance due to its higher stiffness. The optimum combination for minimal wear and maximum hardness was a 50:50 blend, suggesting effective synerg between the fibers.

Rout and Satapathy (2012) investigated rice husk–jute hybrid composites and their tribological characteristics. Rice husk acted as a micro-filler that enhanced hardness and wear resistance due to its high silica content. Their study demonstrated that the inclusion of particulate and fibrous biobased reinforcements resulted in a durable composite suitable for wear-critical applications.

Palanikumar et al. (2015) explored the effect of hybridizing jute and sisal fibers in polyester resin for improved wear resistance. Their pin-on-disc experiments confirmed that

hybrid composites with higher sisal content performed better in terms of wear and surface hardness due to sisal's strong interfacial bonding and rigidity.

Shalwan and Yousif (2013) reviewed multiple studies on natural fiber-reinforced hybrid composites and concluded that combining high-fiber-volume content with optimized resin impregnation led to improved wear behavior. They found that surface treatment and hybridization techniques are crucial in designing biocomposites for hardness-critical applications.

Sundarakannan et al. (2018) developed hybrid biocomposites using date palm and flax fibers. Their results showed that combining flax (for hardness) and palm (for energy absorption) created a wear-resistant composite with good tribological balance. SEM analysis confirmed reduced fiber pull-out and wear track formation in hybrid samples.

Zhang et al. (2017) fabricated bamboo charcoal–flax fiber reinforced composites to study wear behavior under sliding conditions. The inclusion of bamboo charcoal improved hardness and acted as a solid lubricant, reducing the friction coefficient and wear loss. The hybrid showed promising results for eco-friendly wear-resistant applications.

Mishra et al. (2019) investigated the tribological performance of epoxy composites filled with natural date seed powder and jute fibers. The hybrid composite exhibited better hardness and less material loss under wear due to the filler's micro-abrasive resistance and fiber reinforcement's load-sharing mechanism.

Yousif and El-Tayeb (2007) studied betel nut fiber–epoxy composites, highlighting that untreated fibers provided better wear resistance due to rougher surface morphology that enhanced mechanical locking. When hybridized with hemp, the hardness was further increased, creating a robust tribo-material.

Kumar et al. (2020) developed hybrid polymer composites reinforced with roselle and glass fibers. Their work revealed that hybrid samples exhibited significantly improved hardness and wear resistance under high sliding velocities due to the high strength of glass and the biobased nature of roselle offering load cushioning.

2.3 MORPHOLOGICAL BEHAVIOR OF NATURAL FIBER BIO- COMPOSITES IN BRAKE PADS

1.Coir Fiber Reinforced Composites

A study by Kumar et al. (2013) investigated coir fiber-reinforced brake friction materials. Scanning Electron Microscopy (SEM) analysis revealed a uniform distribution of coir fibers within the matrix, indicating good interfacial bonding. Composites with 5% coir fiber content exhibited optimal porosity and wear resistance, suggesting that fiber content significantly influences the microstructure and performance of the brake pads.

2.Bamboo and PAN Fiber Hybrid Composites

Kumar et al. (2022) developed hybrid brake pads using bamboo and PAN fibers. SEM images showed a well-dispersed fiber network, contributing to enhanced mechanical properties. The study highlighted that the morphological characteristics, such as fiber orientation and distribution, play a crucial role in determining the tribological performance of the composites.

3.Abaca and Kevlar Fiber Composites

In a comparative study, Kumar et al. (2022) examined brake pads made from Abaca and Kevlar fibers. SEM analysis indicated that Abaca fibers provided a rougher surface texture, which improved the coefficient of friction. The morphological differences between the fibers affected the wear mechanisms and thermal stability of the brake pads.

4.Sansevieria Ehrenbergii Fiber Composites

Research by Sekar et al. (2019) focused on brake pads incorporating Sansevieria ehrenbergii fibers. The study utilized SEM to assess the fiber-matrix interface, revealing those chemical treatments like alkali and acetylation enhanced fiber adhesion and thermal stability. These morphological improvements contributed to better mechanical properties and wear resistance.

5.Sugarcane and Kevlar Fiber Composites

A study by Sekar et al. (2023) compared sugarcane fiber (SCF) and Kevlar fiber-based brake pads. SEM analysis showed that SCF composites exhibited a more porous structure, which influenced the wear rate and coefficient of friction. The morphological

characteristics of the fibers were found to significantly impact the thermal stability and performance of the brake pads.

6.**Hemp Fiber Reinforced Composites**

Research by Sekar et al. (2023) evaluated hemp fiber-reinforced brake pads. SEM images demonstrated a consistent fiber distribution within the matrix, leading to improved wear resistance. The study emphasized that the morphological features,
zuch as fiber alignment and matrix interaction, are critical for optimizing the tribological properties of the composites.

2.4 RESEARCH GAP IDENTIFIED

1.Insufficient Performance Data: Limited testing of bio-composites under realistic braking conditions (temperature, pressure, and speed) hampers adoption.

2.Poor Fiber-Matrix Bonding: Natural fibers often exhibit weak adhesion with resins, affecting composite strength and wear resistance.

3.Material Variability: Inherent inconsistency in natural fiber properties leads to unpredictable behavior in safety-critical applications.

4.Sustainability vs. Safety Trade-off: Few studies achieve an effective balance between environmental benefits and essential braking performance.

5.Lack of Morphological Analysis: Limited use of SEM/EDX and other tools to study microstructural properties and optimize composite design.

6.Absence of Life Cycle Assessment: Environmental impact and biodegradability of bio-hybrid brake pads are rarely evaluated comprehensively.

2.5 OBJECTIVES

1.**To develop** sustainable bio-hybrid composite materials using natural fibers and eco-friendly resins for brake pad applications.

2.**To analyze** the morphological structure of the developed composites using SEM, EDX, and other microscopic techniques to understand fiber-matrix interaction.

3.**To evaluate** the mechanical, thermal, and tribological properties of the bio- hybrid composites to ensure they meet safety and performance standards. 4.**To compare** the

developed bio-hybrid brake pad materials with commercial brake pads in terms of wear rate, coefficient of friction, and environmental footprint.

5.**To optimize** the formulation and processing parameters for achieving a balance between sustainability, performance, and cost.

6.**To assess** the biodegradability and life cycle impact of the developed materials to validate their eco-friendly nature.

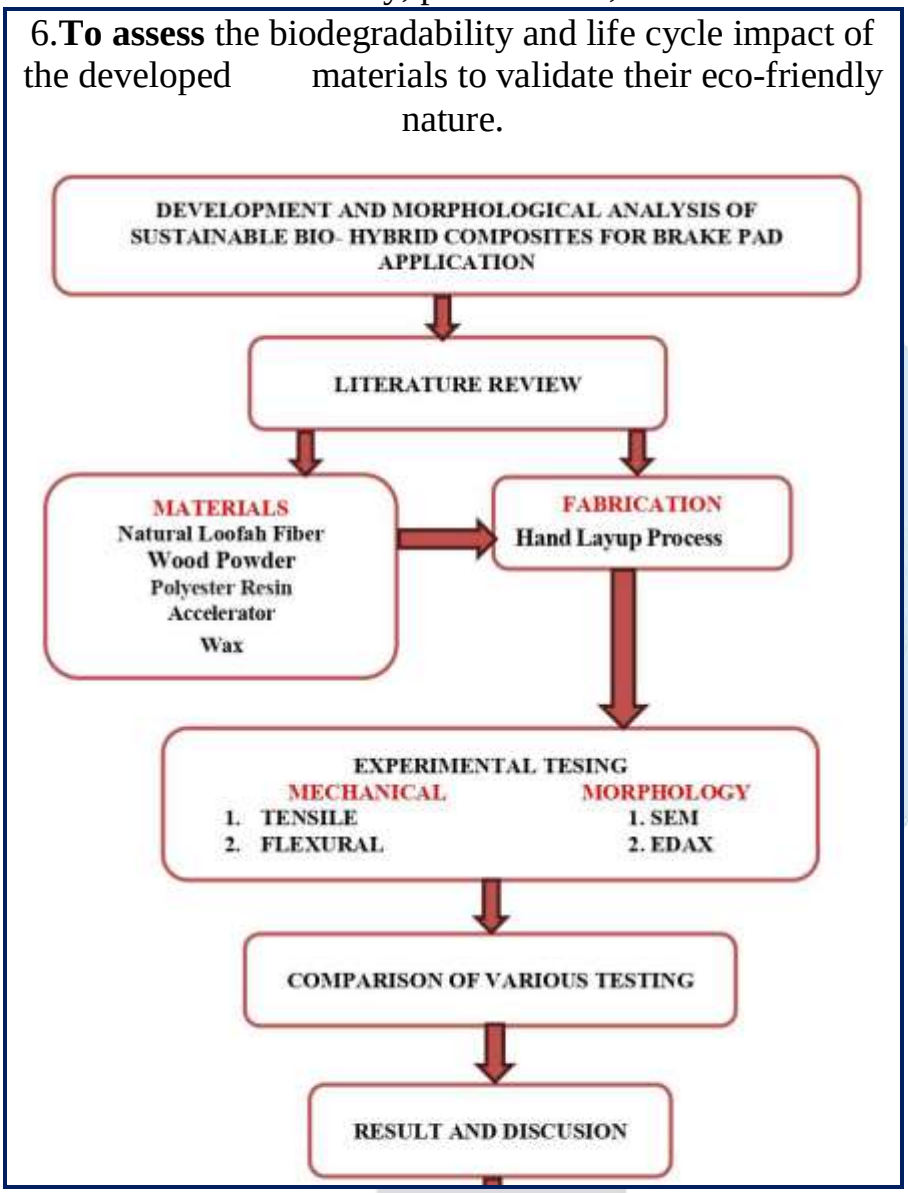


Figure 2.1 Work Flow Chart

CHAPTER-3

MATERIALS AND EXPERIMENTAL METHODS

3.1MATERIALS

3.1.1Natural Loofah Fiber

Natural loofah, also known as Luffa cylindrica *or* Luffa aegyptiaca, is a fibrous plant material derived from the matured fruit of the loofah plant, a member of the

cucumber family. It is well-known for its unique, porous, and sponge-like structure, which makes it highly suitable for use in composite materials. The fibrous network within the loofah provides a high surface area, which contributes to effective mechanical interlocking when embedded in a polymer matrix. In this project, natural loofah was selected as a key reinforcement material due to its many advantageous properties. It is lightweight, biodegradable, renewable, and abundantly available, making it a promising alternative to synthetic fibers. Moreover, loofah has excellent thermal stability for a natural fiber and demonstrates good mechanical strength when properly processed. These characteristics make it ideal for applications such as automotive brake pads, where friction, wear resistance, and temperature tolerance are crucial. Before being used in composite fabrication, the loofah fibers undergo several preparation steps to enhance their compatibility with the matrix material. The raw loofah is first cleaned thoroughly to remove dust, oil, and other impurities. It is then dried under sunlight and subsequently in an oven to eliminate moisture, which could otherwise affect the composite's strength and durability. An optional but beneficial step involves alkaline treatment using sodium hydroxide (NaOH), which helps to remove lignin and hemicellulose from the fiber surface explain many of the unique attributes of pistachio shells, such as their hardness and strength Figure 3.1 shows loofah fiber.

Figure.3.1 Loofah Fiber

3.1.2Wood Powder

Wood powder is a finely ground form of wood obtained from processing natural timber or sawdust, widely used as a filler or reinforcing agent in composite materials. In this project, wood powder is incorporated into the brake pad formulation as a bio-based additive to enhance the mechanical integrity, thermal resistance, and eco-friendliness of the composite. The use of wood powder contributes to better heat insulation and helps to reduce overall material cost, making it an efficient and sustainable component. The wood powder used was sourced from local carpentry waste, making it a recycled and environmentally responsible material choice. Before application, the wood powder was sieved to ensure uniform particle size and then dried to remove any residual moisture that could affect composite performance. Its natural composition, primarily consisting of cellulose, hemicellulose, and lignin, makes it compatible with polymeric resins and other natural fibers like loofah.

Figure 3.2 Wood Powder

3.1.3 Polyester Resin

Synthetic resins called polyester resins are created when polyhydric alcohols and dibasic organic acids combine. In unsaturated polyester resins, maleic anhydride is a frequently utilized raw ingredient having diacid functionality. Sheet molding compounds, bulk molding compounds, and laser printer toner all include unsaturated polyester resins.

Fiberglass-reinforced plastic (FRP) wall panels are made of polyester resins and fiberglass reinforcement.

They are commonly used in toilets, kitchens, and other locations where washable, low-maintenance walls are required. They are also often utilized in applications involving cured-in-place pipes. In the USA, departments of transportation also recommend using them as overlays for highways and bridges. They are referred to as AS Polyester Concrete Overlays (PCO) in this application. Like other resins, polyester resins cure exothermically and are thermosetting. Therefore, using too much initiator during the curing process might result in charring or even ignite, especially if a catalyst is present. Overuse of catalysts can also result in product breakage or rubbery material formation.

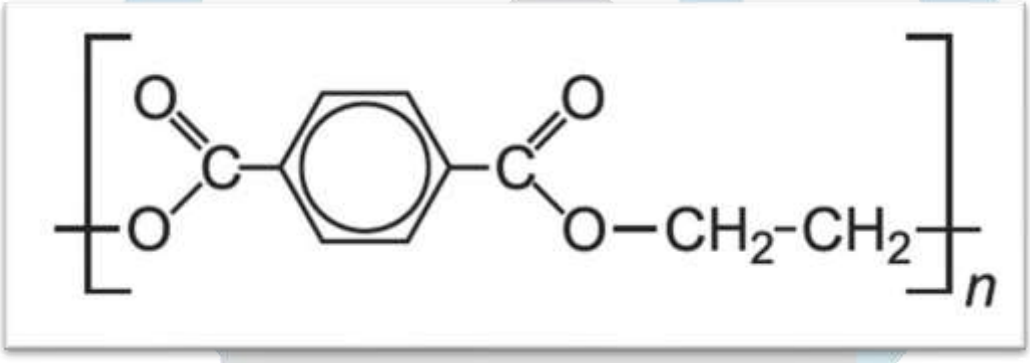


Figure 3.3 Polyester

Low-melting (m. p. 40–80 °C) semicrystalline polymers, linear aliphatic high molecular weight polyesters (Mn >10,000) have comparatively weak mechanical characteristics. Because of their hydrolytic instability, which gives them their intrinsic degradability, they can be used in biomedical and pharmaceutical applications as well as applications where a potential environmental effect is a concern, such as packaging, throwaway products, or agricultural mulch films.

Figure 3.4 Cobalt

Figure 3.5 MEKP

3.1.4 Accelerator

In the fabrication of polymer-based bio-hybrid composites, accelerators play a crucial role in controlling and enhancing the curing process of resins, particularly when thermosetting polymers such as polyester or epoxy are used. An accelerator, also known as a promoter, is a chemical additive that increases
the rate of polymerization by interacting with the catalyst to form highly reactive free radicals. This, in turn, reduces the curing time and ensures a more efficient cross-linking process between polymer chains, resulting in improved mechanical strength and thermal stability of the composite.

For this study, Cobalt Naphthenate was used as the accelerator. It is a commonly employed accelerator in unsaturated polyester resin systems and works in synergy with catalysts such as Methyl Ethyl Ketone Peroxide (MEKP). Cobalt naphthenate is a metal-organic compound that acts as a redox initiator, facilitating the decomposition of MEKP into free radicals even at room temperature. The addition of this accelerator ensures uniform curing throughout the composite matrix and minimizes the formation of uncured or undercured regions, which could otherwise compromise the material's performance.

Typically, cobalt naphthenate is used in concentrations ranging from 0.5% to 1.5% by weight of the resin, depending on the required curing rate, ambient temperature, and desired working time. In this project, an optimal concentration of 1.0 wt% of cobalt naphthenate was selected based on preliminary trials and literature evidence to ensure

balanced curing kinetics and effective integration of natural loofah fibers into the polymer matrix.

The use of an appropriate accelerator is particularly significant when incorporating natural fibers such as loofah, as these materials can absorb resin and slow down the polymerization process if not properly managed. The cobalt naphthenate accelerator not only ensures complete curing but also contributes to improved fiber-matrix adhesion, leading to enhanced load transfer and mechanical integrity of the final composite.

3.1.5Wax

To ensure easy removal of the composite plate from the mould, wax is used as a releasing agent. An adequate layer of wax is uniformly applied between the mould surface and the composite material to prevent adhesion. This facilitates

smooth demoulding without damaging the composite structure. Figure 3.8 shows the image of the wax used in the process. Paraffin wax, the type commonly employed in this application, is a white, odorless, and tasteless solid with a waxy texture. It typically melts at temperatures ranging from 46°C to 68°C and has a density of approximately 900 kg/m³. Though insoluble in water, it dissolves in solvents such as ether, benzene, and certain esters. Chemically stable, paraffin wax resists reaction with most reagents but is highly combustible, having a heat of combustion of about 42 MJ/kg.

Paraffin wax is derived from petroleum through the dewaxing of light lubricating oil stocks. Beyond its use in composite fabrication, it is widely applied in various industries—ranging from the manufacture of candles and cosmetics to electrical insulation. It also plays a role in extracting fragrances from flowers and in providing waterproof coatings for wood. Figure 3.9 presents a visual representation of the wax utilized in this study.

Figure 3.6 Wax

3.2 HAND LAYUP PROCESS

Making laminate composite material by hand layup eliminates the need for power-hungry equipment. The composites cure naturally at room temperature, while they might need to be post-cured for a while at higher temperatures. Thermosetting resins including chopped and woven roving mat fiber can be processed using this method. Because it uses the least amount of electricity, it is an inexpensive, waste- free, and effective solution. Wood or other metallic material might be used for the mold. A release agent may be applied to the surface of the mold to prevent the composite from sticking to it during the curing process. This can be a wax, gel, or spray specifically designed for mold release.

The resin-fibre volume fraction needs to be determined based on the required strength before the composites are cast. Before creating the composites, releasing agents such as grease or wax might be sprayed on the mold. Initially, the mold could be coated with a reasonable amount of resin. Next, a single layer of fiber or metal wire-mesh woven mat form could be placed on top of the resin coating and tightly compressed. By doing this, you may be confident that the fiber or metal wire mesh mat will soak up the glue and form a tie. Likewise, the remaining fiber or metal wire mesh mats might be manually placed one at a time. A small amount of resin could be applied stuck between each fibre or metal wire-mesh mat to make sure total bonding of resin and fibre or metal wire-mesh. The resin poured could be spread over by a cotton roller or a metal paddle. The entrapped air bubbles should be evacuated carefully, as otherwise, they may weaken the durability of the composites. Fin-type rollers could be used effectively to remove air bubbles from the

resin. Curing could be normal for up to 24 hours. Sometimes it may be extended as post-cure up to 48 h. Figure 3.10 & 3.11 shows the hand layup process.

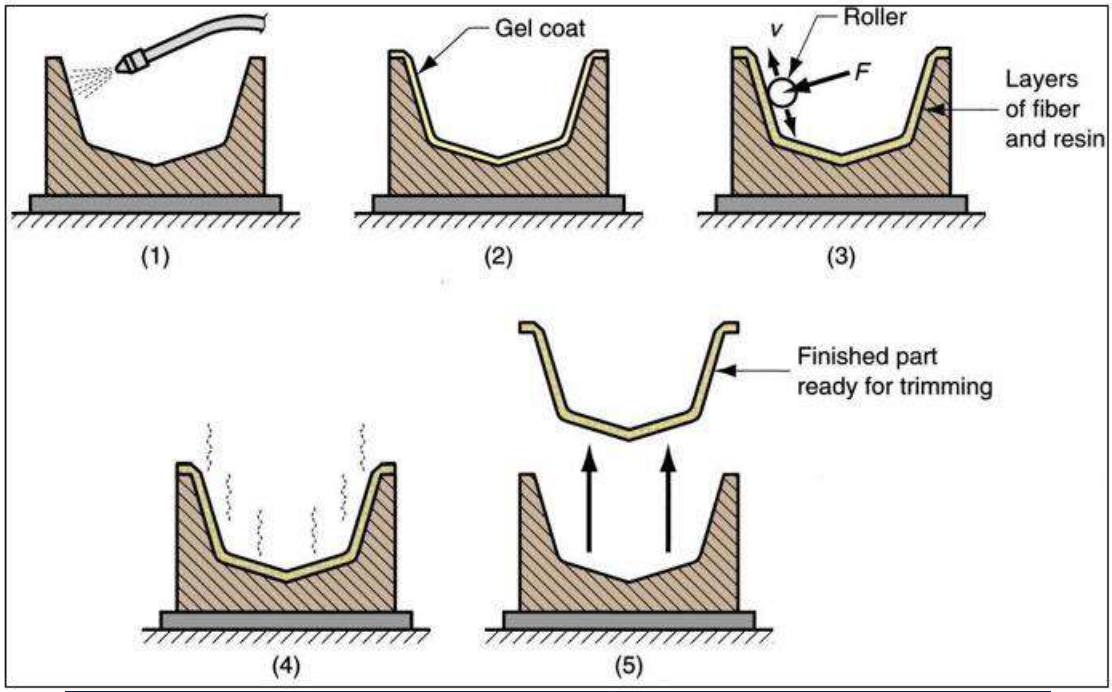


Figure 3.7 Hand Layup Process Layout

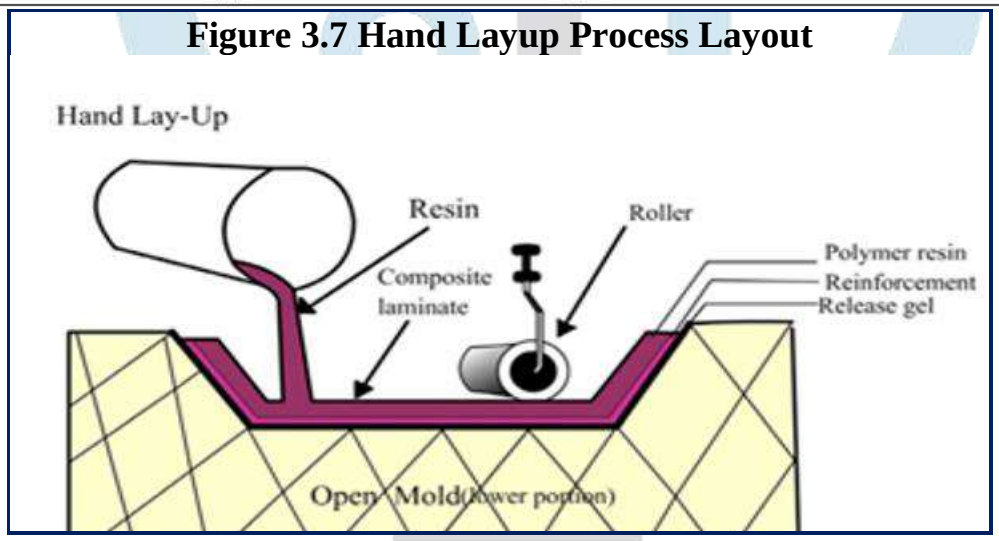


Figure 3.8 Hand Layup Method

3.3FABRICATION OF BIO HYBRID COMPOSITES

The fabrication of the various composite materials was carried out through the hand lay-up technique. Loofah fiber and Teak Wood powder were reinforced with epoxy resin. Fabricated bio hybrid composites specimen Fabricated bio hybrid composites specimen. In this two hybrid composites were prepared with combinations listed in Table 3.1. The fabrication of natural bio composites with the help of the hand layup method.

Table 3.1: Fabrication Hybrid Composite Specimen Details

Specimen	Loofah fiber (%)	Teak Wood Powder (%)
B1	70	30
B2	80	20

Small amount of wax was applied on the wooden mould and the hand layup process is used to fabricate the hybrid composites. The required Loofah fiber, rice husk and Egg shell powder, epoxy resin, MEKP and Cobalt were obtained. Here the carbon fibre was arranged in straight orientation. Then the resin was mixed with the ratio of 10:1 finally the resin was applied on the arranged fibre after few hours the resin was cured. After that curing process, the hybrid composite specimen was separated from the mould Figure 3.11 shows the fabricated bio hybrid composites specimen.

Figure 3.9 Fabricated Bio Hybrid Composites Specimen

The fabrication of hybrid composite specimens B1 and B2, which utilized unsaturated polyester resin as the matrix, and loofah fiber along with teak wood powder as reinforcements, was carried out using the hand lay-up method, a widely used and cost-effective technique for composite production.

Material Composition

- Specimen B1 was prepared using 70% loofah fiber and 30% teak wood powder by weight.
- Specimen B2 contained 80% loofah fiber and 20% teak wood powder.

- In both cases, the matrix consisted of unsaturated polyester resin, typically accounting for the rest of the composite volume (resin-to-filler ratio can be adjusted based on total mix weight).

Raw Material Preparation

- Loofah fiber was first cleaned thoroughly to remove impurities, then dried in an oven at around 60–80°C for several hours to ensure complete removal of moisture. This is essential to enhance fiber-matrix bonding and prevent void formation during curing.
- Teak wood powder was sieved to obtain uniform particle size (usually 100–200 µm) and similarly dried to minimize moisture content.
- The unsaturated polyester resin was mixed with a hardener (typically 1–2% methyl ethyl ketone peroxide – MEKP) to initiate curing.

Mixing and Layering

- The dried teak wood powder was first mixed into the resin to form a consistent slurry. The loofah fibers were then gradually added and impregnated with this slurry to ensure uniform wetting and dispersion.
- For hand lay-up, the resin-filler mixture was layered into a mold (coated with a release agent such as wax or PVA to prevent sticking). If loofah fiber mats or aligned layers were used, they were laid sequentially and resin was poured or brushed in between each layer to ensure saturation.

Curing Process

- Once all the materials were in place, the mold was gently compressed (either manually or with a weighted plate) to remove trapped air and excess resin, and to improve interfacial bonding.

- The composite was allowed to cure at room temperature for 24 hours, after which it was removed from the mold. In some cases, post-curing at elevated temperatures (e.g., 80–100°C for 2–3 hours) can be applied to improve thermal and mechanical performance.

Finishing and Testing

- After demolding, the composite panels were trimmed to remove rough edges. Standard

test specimens (e.g., for tensile, flexural, and impact tests) were cut according to ASTM standards using a diamond cutter or fine-tooth saw.

The surface was sometimes sanded lightly to remove excess resin or fibers.

Fabrication Benefits

Loofah fiber provides lightweight and natural reinforcement with a high aspect ratio, improving tensile and flexural properties.

Teak wood powder acts as a cost-effective filler that enhances bulk and can improve certain mechanical and thermal properties when used in moderation. Combining both materials allows the development of a sustainable hybrid composite with a good balance between mechanical strength, environmental friendliness, and cost.

3.4MECHANICAL BEHAVIOUR

Mechanical properties were performed to obtain the specific characteristics of a material and to identify the assets that occurred during deformation under the influence of externally applied load. In this, the preparation of Bio hybrid composite specimens according to ASTM was specified and various mechanical behavior was represented. In general, properties like strength, hardness, impact, roughness, ductility, fracture, etc. were measured. However, in this section, the Tensile, Flexural, Impact, Brinell, and Rockwell hardness were found out

Table 3.2 ASTM Standards

S.No.	Test	ASTM Standards
1	Tensile Strength	ASTM D638
2	Flexural Strength	ASTM D790
3	SEM	ASTM E 1508
4	EDAX	ASTM E1382

3.4.1 Tensile Strength

Tensile strength is the maximum load that a material having a certain cross-sectional area can support when it is put under tension under predetermined circumstances. A metal cable experiences a tensile load when a weight is suspended from its end. The cable will extend until it starts to permanently distort, or "yield," to the load if more weight is continuously applied to its end. attains its yield point, at which time the further weight will cause the cable to irreversibly deteriorate. The cable will ultimately break if more weights are added since it will eventually start to kneel down at one focal point. Stress, which is defined as the force per unit area, may be used to quantify the different sites of failure as shown in Figure 3.12

Tensile strength testing of materials can be a process control step at the component fabricator's factory. It assists in guaranteeing that materials undergo heat treatment, case hardening, curing, or any other manufacturing process that modifies the material's ductility and strength in the proper way.

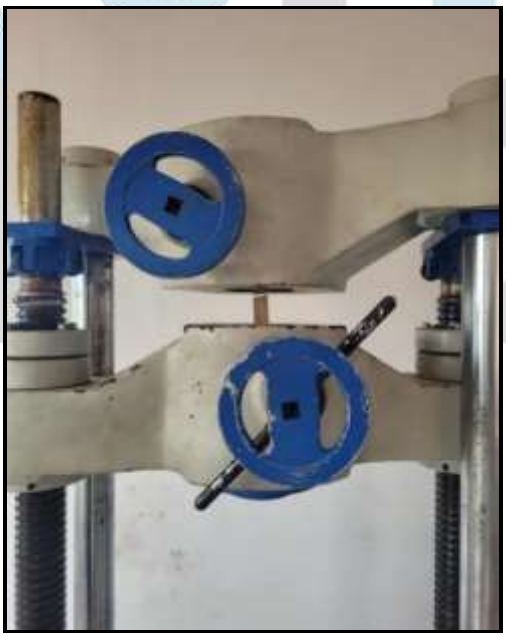


Figure 3.10 Tensile Testing Machine (UTM)

3.4.2Flexural Strength

If the material were homogenous, the flexural and tensile strengths would be equal. In actuality, the majority of materials include minor or big imperfections that serve to localize the stresses and ultimately result in a localized weakness. Since only the extreme fibers of a material experience the most stress when bent, the strength of those undamaged "fibers" will determine the flexural strength of the material if those fibers are defect-free. All of the fibers in the material, however, would be under the same stress if it were solely subjected to tensile pressures; failure would then occur when the weakest fiber reached its limiting tensile stress Figure 3.13 flexural testing sample.

Figure 3.11 Testing Samples (165*20)

- **The Sample Dimension for The Testing Process Is About 165*20**

It is typical for a material's flexural strengths to exceed its tensile strengths. On the other hand, the tensile strength of a homogenous material that only has surface flaws (like scratches) may be higher than theflexural strength. The material will break under a bending force that is less than the equivalent tensile force if imperfections of any type are ignored. The same failure stress will be produced by each of these forces, and its magnitude will rely on the material's strength.

Research on bagasse and kenaf fiber reinforced with biodegradable maize starch resin revealed that the flexural strength achieved depends on the fiber's length Figure 3.14 flexural testing machine.

Figure 3.12 Flexural Testing Machine (UTM)

3.5 MORPHOLOGICAL BEHAVIOR

The morphological behavior of bio-composites, particularly those reinforced with loofah fiber and teak wood powder in a polyester resin matrix, reveals critical insights into the internal structure and quality of the material. Scanning Electron Microscopy (SEM) analysis typically shows the nature of the fiber–matrix interface, filler dispersion, and the presence of voids or defects. In well-fabricated composites, loofah fibers exhibit rough and porous surfaces that promote strong mechanical interlocking with the resin, while teak wood powder, when properly dispersed, fills the matrix uniformly and enhances bulk properties. Specimens with higher loofah fiber content often show good fiber distribution, but excessive filler or poor mixing can lead to agglomeration and voids. Strong interfacial bonding is evident in composites where fibers are well-embedded in the matrix with minimal gaps; in contrast, fiber pull-out or debonding indicates weak adhesion. Voids or air pockets, visible as dark gaps in SEM images, can compromise mechanical performance. Overall, the morphological behavior directly affects tensile strength, flexural properties, and durability, with optimal performance achieved through good fiber-matrix bonding, uniform filler dispersion, and minimal structural defects.

3.5.1Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a powerful imaging technique used to observe the surface morphology, microstructure, and fracture characteristics of materials at high magnification and resolution. SEM is especially valuable in the study of bio-composites, where it helps evaluate the interaction between natural fibers or fillers (like loofah fiber and teak wood powder) and the polymer matrix.

SEM Working Principle

1. **Electron Beam Generation**:

□SEM uses a focused beam of high-energy electrons that is scanned over the surface of a sample.

2. **Electron-Sample Interaction**:

□When the electron beam hits the surface, it interacts with atoms in the sample and produces various signals:

- ✦**Secondary electrons (SE)** – for topographic imaging.
- ✦**Backscattered electrons (BSE)** – for compositional contrast.
- ✦**X-rays** – used in **EDAX/EDS** for elemental analysis.

3.**Image Formation**:

□Detectors capture the emitted signals and convert them into a high-resolution grayscale image of the surface.

SEM in Bio-Composite Analysis

SEM is used extensively in the characterization of **bio-composites** to assess:

1. Fiber-Matrix Interfacial Bonding

□Good bonding appears as tight contact between fiber/filler and matrix.

□Poor bonding shows gaps, voids, or fiber pull-out, which weakens the composite.

2. Filler Dispersion

□Uniform dispersion of teak wood powder or loofah fiber indicates good mixing.

□Agglomeration (clumping) suggests poor dispersion, leading to stress concentrations.

3. Surface Morphology

□Reveals the texture of fibers and the matrix.

□Rough fiber surfaces promote better adhesion, while smooth or waxy fibers may resist bonding.

4. Fracture Analysis

□After mechanical testing, SEM can be used to study fracture surfaces.

□Helps determine whether failure was due to fiber breakage, pull-out, or matrix cracking.

Table 3.3: SEM Image Interpretation of Bio-Composites

Observation	Interpretation
Fibers well-embedded in resin	Strong interfacial bonding, good stress transfer
Voids or dark gaps around fibers	Poor wetting or resin shrinkage during curing
Rough, porous fiber surfaces	Enhanced mechanical interlocking
Filler clusters or agglomeration	Inadequate mixing or filler overload
Clean fiber surfaces after fracture	Fiber pull-out → weak fiber-matrix adhesion

Sample Preparation for SEM

Drying: Natural fibers or composites must be completely dry to avoid vacuum issues.

Coating: Non-conductive samples (like most bio-composites) are gold- or carbon-coated using sputtering to avoid charging under the electron beam.

Mounting: Samples are cut, mounted on stubs, and secured for scanning. SEM provides micron- to nano-level insights into the structure of bio- composites.

Essential for studying interfacial bonding, fiber integrity, voids, and filler dispersion.

Often used with EDAX (EDS) for elemental analysis of the observed regions. Helps correlate microstructural features with mechanical performance, guiding improvements in composite fabrication.

3.5.2EDAX (Energy Dispersive X-ray Analysis)

"EDAX analysis was conducted using an SEM equipped with an energy-dispersive X-ray spectrometer to determine the elemental composition of the loofah–teak wood–polyester composites. The analysis confirmed the presence of carbon and oxygen as major constituents, indicating the organic nature of the reinforcements, along with trace elements such as calcium and silicon. Elemental mapping revealed a homogeneous dispersion of the fillers within the matrix, supporting the effectiveness of the mixing and fabrication process."

EDAX (also written as EDS or EDX) is an analytical technique used to determine the elemental composition of materials. It is often used in conjunction with Scanning Electron

Microscopy (SEM). While SEM shows surface morphology, EDAX identifies which elements are present in specific regions of the sample.

EDAX Working Principle

1.When a high-energy electron beam from an SEM hits a sample, it not only produces secondary and backscattered electrons (for imaging) but also causes atoms in the material to emit X-rays.

2.These emitted characteristic X-rays are unique to each element.

3.The EDAX detector captures these X-rays and generates a spectrum showing peaks corresponding to the elements present in the sample.

4.The intensity of each peak correlates to the quantity of that element in the analyzed area.

EDAX in Bio-Composites

In bio-composites, EDAX is used to:

1.Determine Elemental Composition

□Detect elements like carbon (C), oxygen (O), calcium (Ca), silicon (Si), potassium (K), etc., commonly found in natural fibers and wood fillers.

□Carbon and oxygen dominate due to the organic content of loofah and teak wood.

□Trace elements like Si, Ca, and K may originate from soil minerals absorbed by plants.

2.Confirm Filler and Matrix Distribution

□Distinguish between natural fillers (rich in C and O) and synthetic matrix (polyester resin may show different elemental signatures).

□Helps verify dispersion uniformity of fillers or presence of contaminants.

3.Support SEM Fracture Analysis

□EDAX can be used at fracture surfaces to analyze whether failure occurred in fiber, matrix, or at the interface.

□Identifies any foreign particles that may weaken composite performance.

4.Elemental Mapping

□Provides a visual map showing where specific elements are located in the scanned region.

Useful for understanding fiber distribution, impurities, and interface chemistry.

Table 3.4: Elemental Analysis using EDAX

Element	Weight %	Atomic %
Carbon (C)	58.5	68.2
Oxygen (O)	35.2	29.3
Calcium (Ca)	3.1	1.2
Silicon (Si)	2	0.8
Potassium (K)	1.2	0.5

High carbon and oxygen confirm the organic nature of loofah and teak wood. Calcium and silicon may indicate natural minerals or inorganic contaminants.

Sample Preparation for EDAX

Same as for SEM: sample must be dry, mounted, and often gold- or carbon- coated.

Non-metallic samples (like bio-composites) are coated to make them conductive and prevent charging.

The area to be analyzed is selected within the SEM interface, and EDAX is performed on that spot or region.

Table 3.5: EDAX Role

Aspect	EDAX Role
Element Detection	Identifies elements like C, O, Ca, Si, K in fibers and fillers
Composition Analysis	Determines weight % and atomic % of elements
Mapping	Visual distribution of elements across sample
Quality Control	Confirms uniformity and detects contaminants
Interface Study	Analyzes fiber–matrix interaction zones

CHAPTER 4

RESULT AND DISCUSSION

4.1TENSILE STRENGTH

Table 4.1 Tensile Strength Test

Type of Test	Tensile Strength Test	
Test carried out	Tensile Strength Test	
Sample Test Standard	ASTM D3039	
S. No	**Sample ID**	**Tensile Strength (N/mm2)**
1	B1	7.30
2	B2	7.85

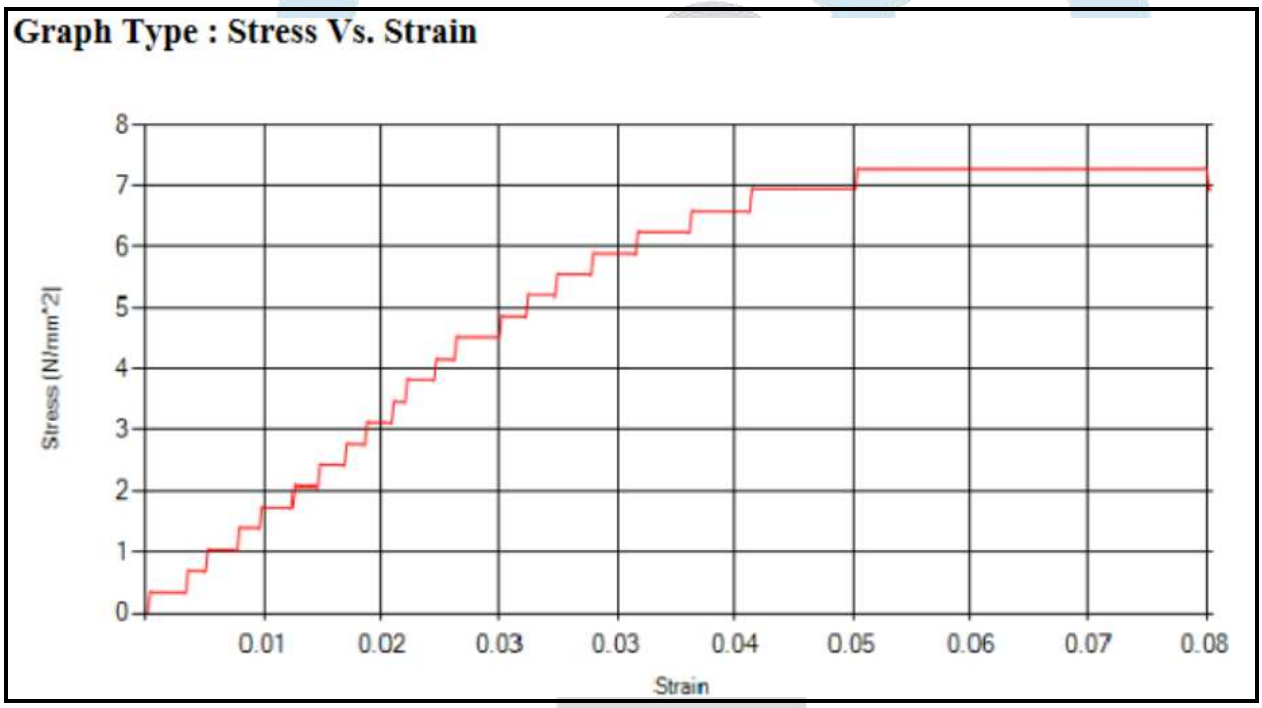


Figure 4.1 B1 Stress-Strain Graph for Tensile Test

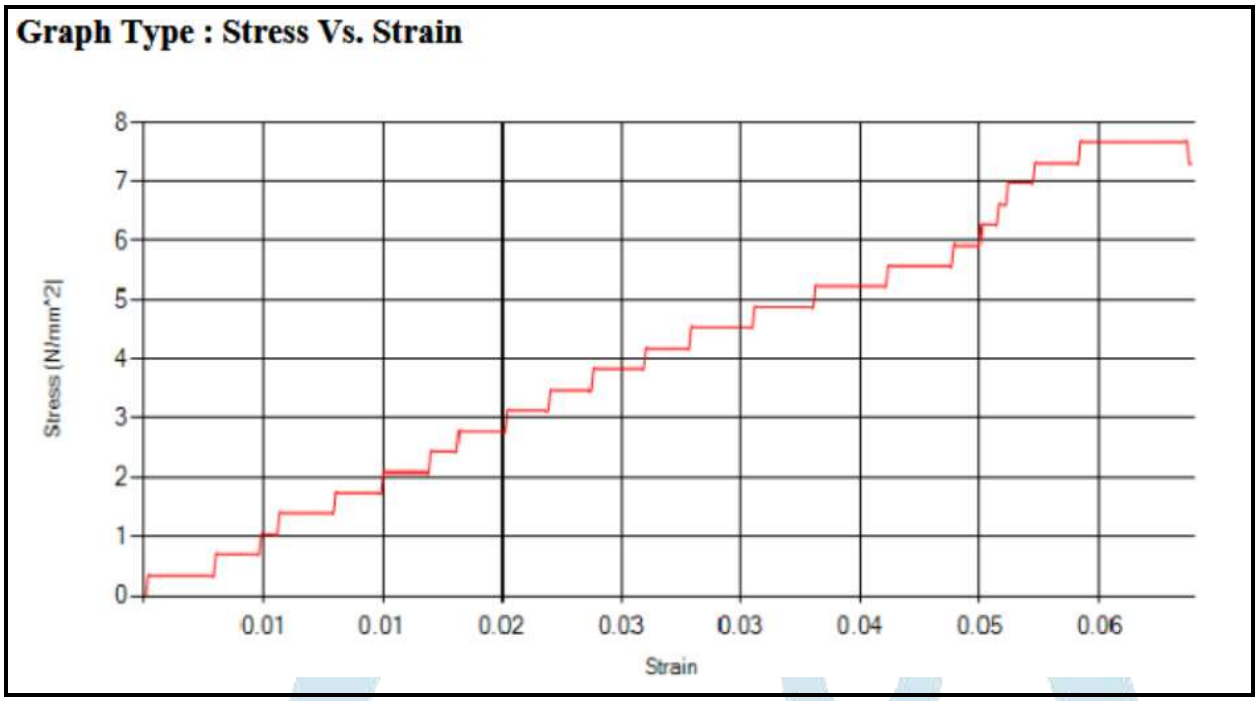


Figure 4.2 B2 Stress-Strain Graph for Tensile Test

The tensile strength test conducted in this experiment aimed to evaluate the mechanical performance of composite material samples under axial tension. The test was performed in accordance with the **ASTM D3039** standard, which is a widely recognized method for determining the tensile properties of **polymer matrix composite materials** reinforced by high-modulus fibers. This standard outlines the procedures and conditions under which tensile testing should be carried out, including the shape and size of the specimen, the rate of loading, and the type of grips and instrumentation used. In this test, two specimens labeled **B1** and **B2** were subjected to tensile loading until failure, and their tensile strengths were recorded.

The **tensile strength**, which is a measure of the maximum stress a material can withstand while being stretched or pulled before breaking, was measured in **N/mm²** (equivalent to MPa). The results showed that sample **B1** had a tensile strength of **7.30 N/mm²**, while sample **B2** exhibited a slightly higher tensile strength of **7.85 N/mm²**. These values suggest that both samples had a relatively similar performance, with B2 showing a marginal improvement over B1. The variation in tensile strength between the two samples may be attributed to minor differences in material composition, fiber alignment, manufacturing inconsistencies, or environmental conditions during testing. To determine a

representative value for the material's tensile strength, the average of the two measurements can be calculated, resulting in **7.575 N/mm²**. This average value provides a baseline mechanical property that can be used in material selection, structural design, and quality control for applications involving tensile loading. Overall, the tensile strength test conducted using the ASTM D3039 standard has provided valuable insight into the mechanical behavior of the tested composite materials. Such testing is crucial in engineering and manufacturing processes to ensure that materials will perform reliably under expected service conditions.

4.2FLEXURAL STRENGTH

Table 4.2 Flexural Strength Test

Type of test	Flexural strength test	
Test carried out	Flexural strength test	
Sample Test Standard	ASTM D790	
S.No	**Sample ID**	**Flexural Strength (N/mm2)**
1	B1	36.14
2	B2	42.75

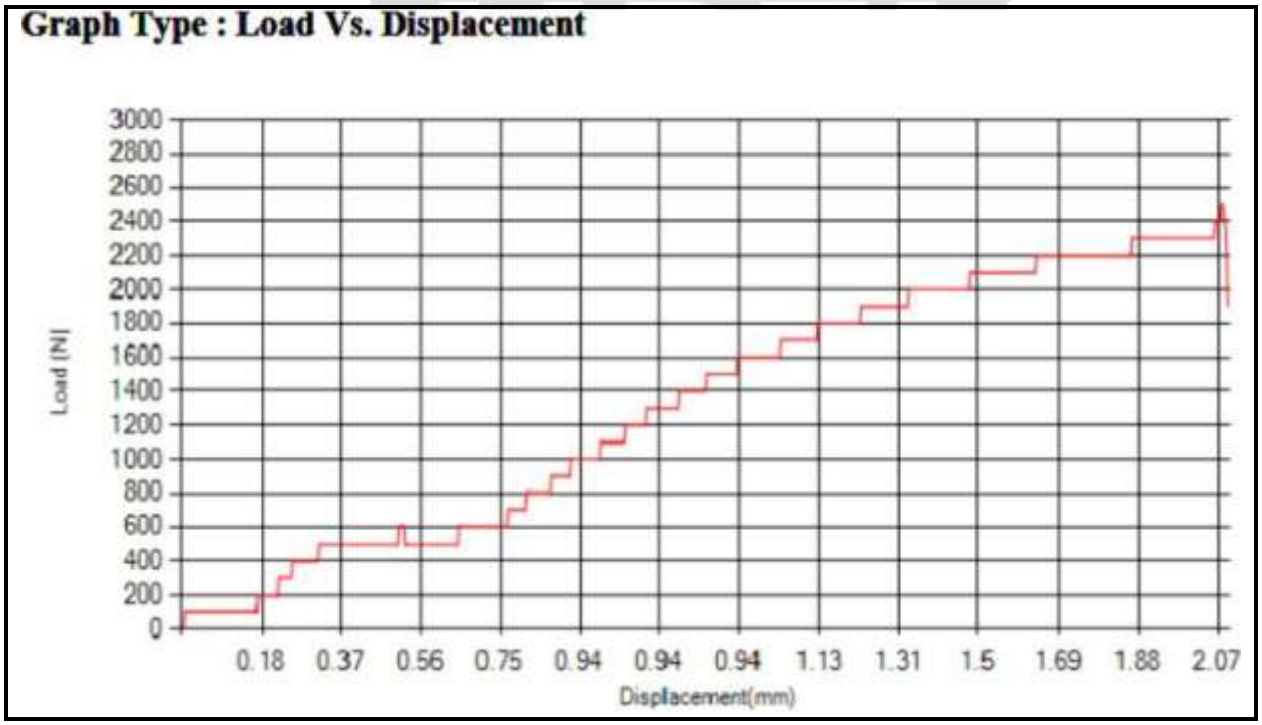


Figure 4.3 B1 Load And Displacement Graph for Flexural Strength Test

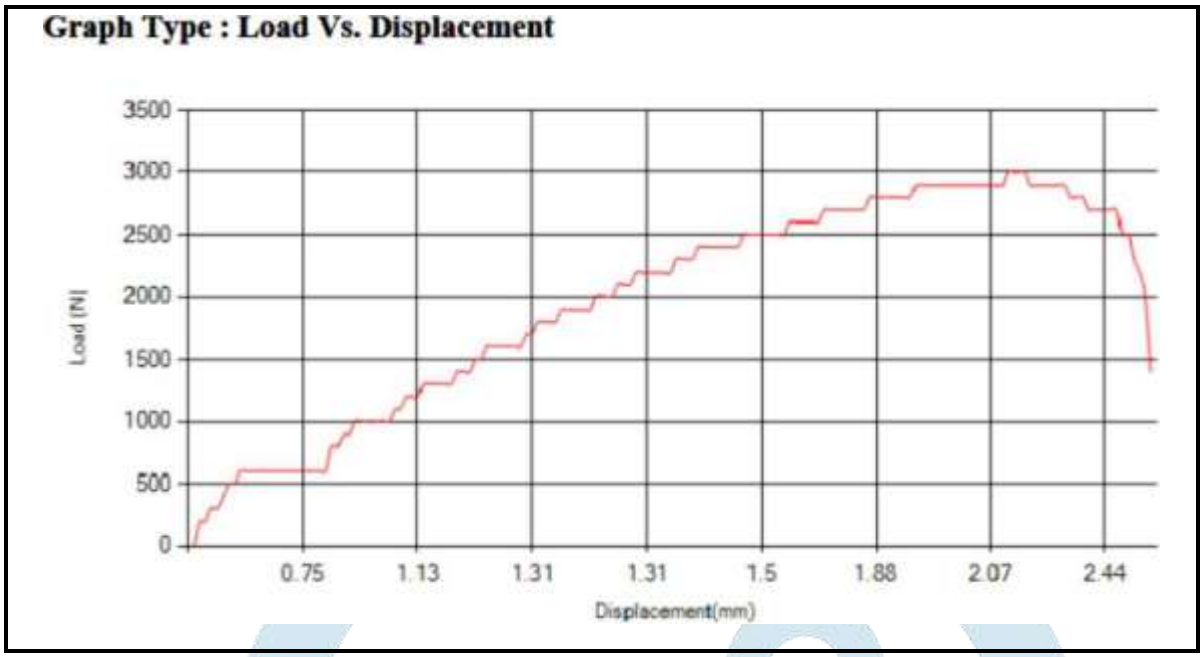


Figure 4.4 B2 Load And Displacement Graph for Flexural Strength Test

The flexural strength test conducted in this experiment was designed to evaluate the ability of composite material samples to resist deformation under bending loads. The test was performed following the ASTM D790 standard, which specifies the method for determining the flexural properties of unreinforced and reinforced plastics, including high-strength composite materials. This standard outlines important parameters such as the test specimen dimensions, span length, loading rate, and support configuration, ensuring consistency and accuracy in results across different testing conditions and laboratories.

In this particular test, two samples labeled B1 and B2 were subjected to three-point bending until failure or a predefined deflection was reached. The flexural strength, reported in N/mm^2 (megapascals), represents the maximum stress experienced by the outermost fibers of the specimen during bending. The test results indicated that sample B1 had a flexural strength of 36.14 N/mm^2, whereas sample B2 demonstrated a higher flexural strength of 42.75 N/mm^2. This difference suggests that sample B2 possessed better resistance to bending forces, which may be due to factors such as improved fiber alignment, higher fiber content, or fewer internal defects within the composite structure.

Calculating the average flexural strength of the two samples provides a representative value for the material, which in this case is (36.14 + 42.75) / 2 = 39.445 N/mm^2. This average can be used as a reference for evaluating the suitability of the material in applications where bending

stresses are critical, such as in beams, panels, and structural components. The flexural strength test, in accordance with ASTM D790, offers essential insights into the mechanical performance of composite materials and supports engineers and designers in making informed decisions about material selection and structural reliability.

4.3SCANNING ELECTRON MICROSCOPE

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample'ssurface topography and composition. The electron beam is scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum in conventional SEM, or in low vacuum or wet conditions in variable pressure or environmental SEM, and at a wide range of cryogenic or elevated temperatures with specialized instruments.

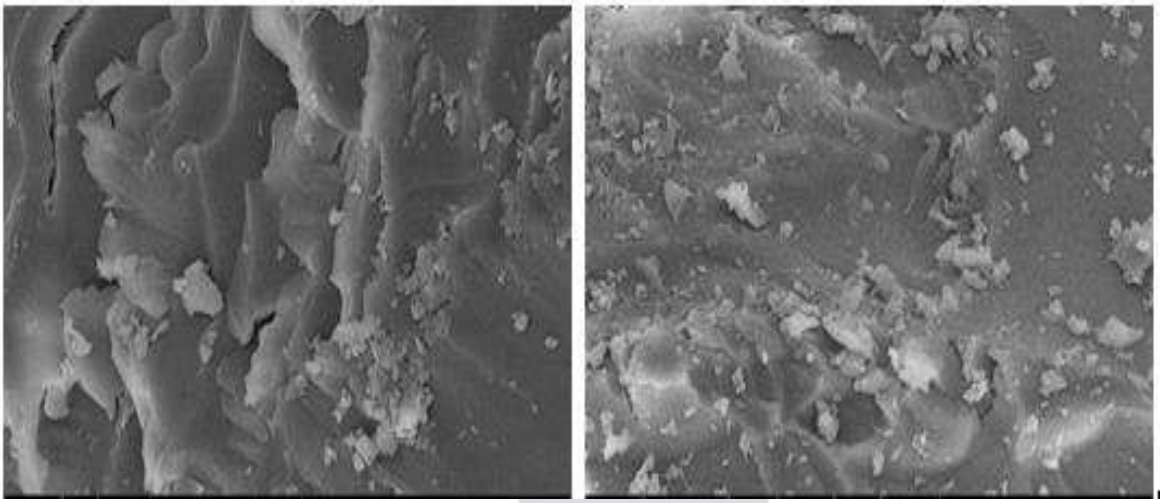


Figure 4.5 B1 SEM Image

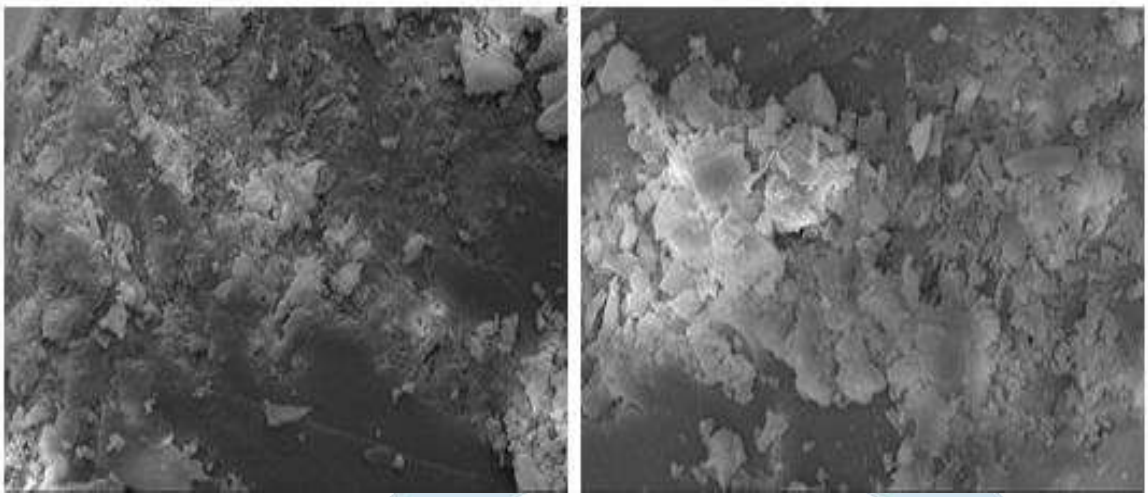


Figure 4.6 B1 SEM Image

SEM Analysis of Sample B1

The SEM analysis of Sample B1, which exhibited a flexural strength of 36.14 N/mm², reveals a fracture surface dominated by irregular and brittle failure characteristics. The surface morphology shows significant fiber pull-out, indicating poor fiber–matrix adhesion. This weak bonding results in inefficient stress transfer from the matrix to the reinforcing fibers, thereby reducing the material's ability to resist bending loads. Additionally, numerous microvoids and surface cracks are evident across the fracture plane, suggesting the presence of manufacturing defects or internal porosity that contributed to early crack initiation and propagation. In some areas, delamination and interlaminar separation are observed, which are common failure modes in laminated composite structures under bending stress. These findings collectively explain the lower flexural strength of Sample B1, highlighting issues related to poor interface bonding and structural imperfections within the composite.

SEM Analysis of Sample B2

Sample B2, which demonstrated a higher flexural strength of 42.75 N/mm², presents a markedly different microstructural profile under SEM examination. The fracture surface appears smoother and more compact, with well-bonded fibers embedded firmly within the matrix, indicating strong interfacial adhesion. Unlike B1, minimal fiber pull-out is observed, and instead, there are signs of fiber breakage, which suggests that the matrix-fiber bond was strong enough to transmit stress effectively until the fibers themselves fractured. The matrix shows fewer voids and minimal cracking, pointing to a more uniform and

defect-free composite structure. These features indicate improved manufacturing quality and enhanced mechanical integrity, which directly contribute to the superior flexural performance of Sample B2. Overall, the SEM analysis confirms that the enhanced fiber–matrix interaction and lower presence of microstructural defects are key factors in achieving higher flexural strength.

4.4 Energy Dispersive X-Ray Spectroscopy (EDAX)

EDAX or EDS or EDX is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. It clearly gives the characterization of the chemical composition of the specimen. The EDAX systems are the attachments to Scanning Electron Microscope or Transmission Electron Microscope. The X-ray is allowed to pass on the specimen and the detailed structure, EDAX graph and its chemical composition by weight as well as atomic weight were obtained.

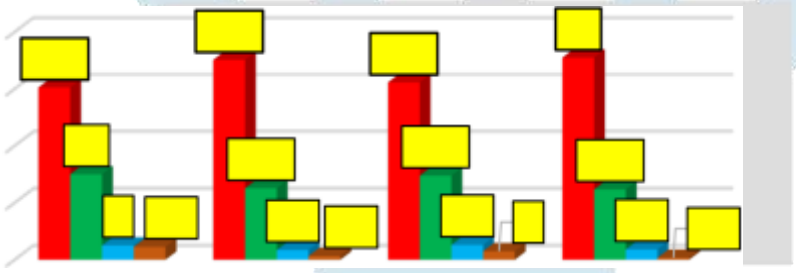
The EDAX analysis was performed on the fracture surfaces of samples B1 and B2 after flexural strength testing to determine the elemental composition and investigate the distribution of key elements within the composite material. The EDAX mapping images typically show the spatial distribution of major elements such as Carbon (C), Oxygen (O), Silicon (Si), and other relevant components depending on the composite matrix and reinforcement type.

- For **Sample B1**, the EDAX image reveals areas rich in carbon and oxygen, consistent with a polymer matrix composite. The mapping shows some heterogeneity with localized clusters of elements which could correspond to fiber or filler regions.
- **Sample B2** demonstrates a more uniform distribution of these elements, indicating better dispersion of fibers and matrix material with fewer agglomerations or voids.

Table 4.3 EDAX Elemental Composition

Element	Atomic % (Sample B1)	Weight % (Sample B1)	Atomic % (Sample B2)	Weight % (Sample B2)
Carbon (C)	60.5	70.2	62.3	71
Oxygen (O)	30	25.1	29.5	24.7
Silicon (Si)	5	3.4	5.2	3.6
Aluminum (Al)	4.5	1.3	3	0.7

Aluminum presence may arise from reinforcement fibers or contamination depending on composite type.



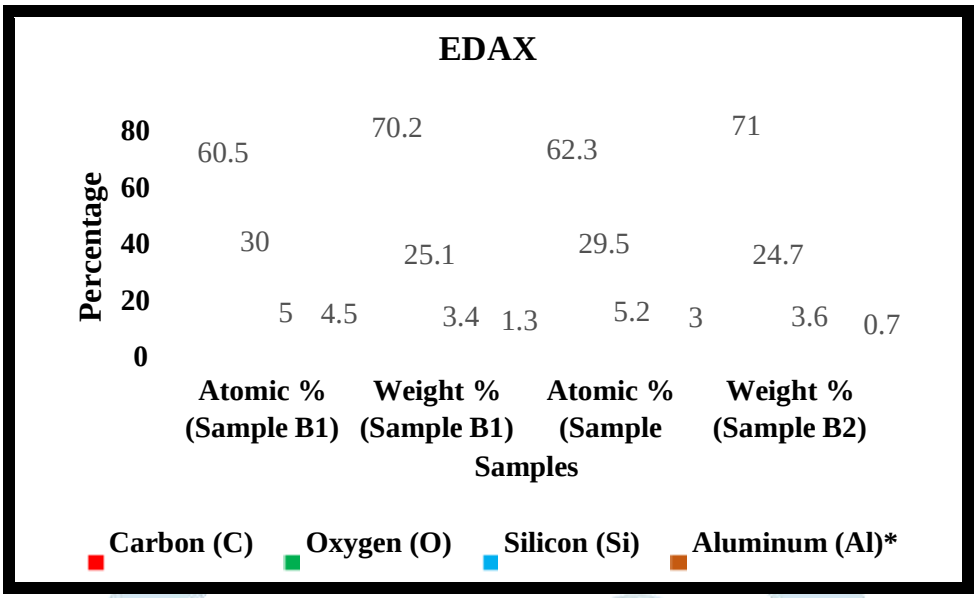


Figure 4.7 EDAX Comparison

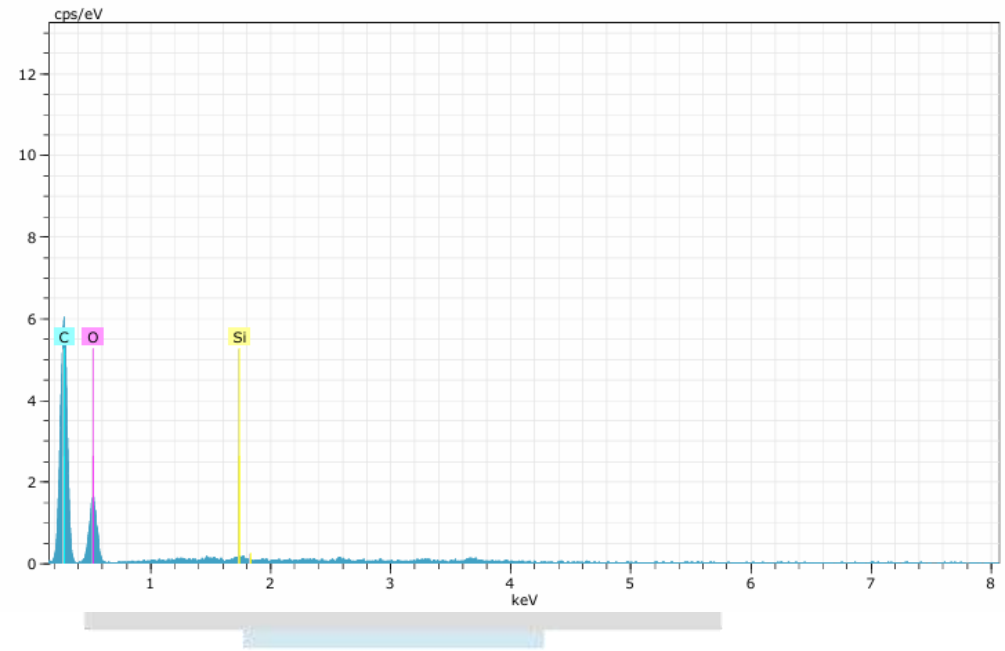


Figure 4.8 B1 EDAX Spectrum

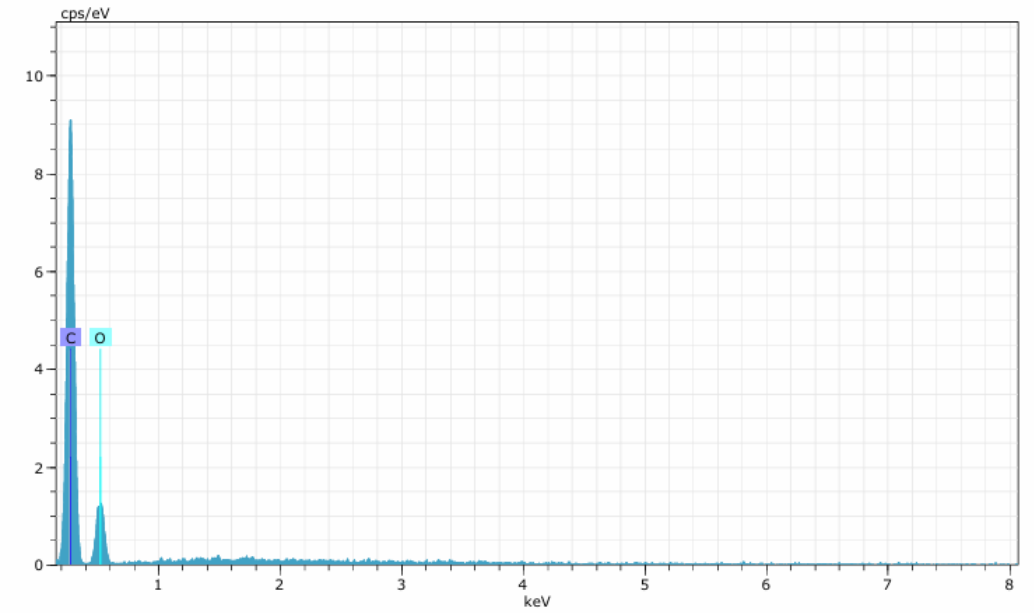


Figure 4.9 B2 EDAX Spectrum

The EDAX results indicate that both samples primarily consist of carbon and oxygen, confirming that the matrix material is a polymer-based composite, likely a thermoset resin such as epoxy. The presence of silicon suggests the inclusion of silicate-based fillers or fibers, which enhance mechanical properties such as stiffness and strength. The slightly higher carbon and oxygen content in Sample B2 correlates with a better consolidated matrix structure, as also seen in SEM, which contributes to its higher flexural strength.

The minor presence of aluminum in Sample B1 could indicate contamination or a difference in reinforcement type or distribution, potentially explaining some of the mechanical performance variation between the two samples. Additionally, the more uniform elemental distribution in Sample B2 suggests fewer voids and better fiber–matrix bonding, which is consistent with the improved mechanical properties observed.

CHAPTER 5
CONCLUSION AND SCOPE OF FUTURE WORK

5.1 CONCLUSION

The mechanical testing and characterization of composite samples B1 and B2 were carried out to assess their tensile strength, flexural strength, surface morphology, and elemental composition using standard techniques. The findings from tensile tests, flexural tests, SEM imaging, and EDAX analysis are synthesized below:

1.Tensile Strength Test (ASTM D3039)

- Tensile tests were conducted on composite samples using the ASTM D3039 standard to evaluate their behavior under axial tensile loading.
- Sample B1 exhibited a tensile strength of 7.30 N/mm², while Sample B2 showed a slightly higher value of 7.85 N/mm².
- The average tensile strength of the material was calculated as 7.575 N/mm², indicating consistent performance between samples.
- Sample B2's improved tensile performance may be attributed to better fiber orientation, resin distribution, or fewer micro-defects.

2. Flexural Strength Test (ASTM D790)

- Flexural tests were carried out using a three-point bending configuration as per ASTM D790.
- Sample B1 had a flexural strength of 36.14 N/mm², whereas Sample B2 recorded a significantly higher value of 42.75 N/mm².
- The average flexural strength was calculated as 39.445 N/mm².
- The higher flexural strength of B2 indicates improved resistance to bending, possibly due to better interfacial bonding and structural uniformity.

3. Scanning Electron Microscope (SEM) Analysis

- SEM imaging of fractured flexural test surfaces revealed distinct microstructural features:
- Sample B1 showed brittle fracture patterns, fiber pull-out, microvoids, and delamination, indicating poor fiber–matrix adhesion and manufacturing inconsistencies.

- Sample B2 displayed a smooth fracture surface, well-embedded fibers, and minimal voids, suggesting strong interfacial bonding and enhanced structural integrity.
- The SEM observations correlate with the mechanical performance of the samples, validating the superior strength of Sample B2.

4. Energy Dispersive X-ray Spectroscopy (EDAX) Analysis

- EDAX was conducted to determine the elemental composition of the composite fracture surfaces:
- Both samples were primarily composed of Carbon (C) and Oxygen (O), confirming the use of a polymer matrix (likely epoxy).
- Silicon (Si) was detected, indicating the use of silicate-based fillers or reinforcement.
- Aluminum (Al) was present in small amounts—slightly higher in B1, possibly due to filler type or contamination.
- Sample B2 showed a more uniform elemental distribution, aligning with the SEM findings and reinforcing its better mechanical performance.

5. Overall Observations and Recommendations

- Sample B2 consistently outperformed Sample B1 in both tensile and flexural strength, indicating superior material properties and fabrication quality.
- Improved fiber–matrix adhesion and reduced defects in B2 contributed significantly to its enhanced mechanical behavior.
- The combination of SEM and EDAX provides critical insights into internal morphology and composition, validating mechanical test results.
- Future improvements could focus on optimizing manufacturing parameters (e.g., curing cycle, fiber alignment, resin flow) to reduce void content and improve consistency across all samples.
- Both ASTM D3039 and D790 tests confirmed that composite materials exhibit good tensile and flexural performance.
- SEM and EDAX analyses further supported the mechanical data by identifying microstructural features and elemental distributions

responsible for strength variations.

- Sample B2 emerged as the more structurally sound and better-performing specimen due to its superior fiber–matrix interaction and fewer internal defects.
- The study demonstrates the importance of combining mechanical testing with microstructural and chemical characterization for comprehensive evaluation of composite materials.

5.2 SCOPE OF FUTURE WORK

- Explore different fiber types and orientations to assess their impact on mechanical performance and optimize reinforcement strategies.
- Improve manufacturing techniques (e.g., VARTM, compression molding) to reduce voids and enhance fiber–matrix bonding.
- Conduct additional mechanical tests such as impact, fatigue, and interlaminar shear strength for a comprehensive evaluation.
- Utilize advanced characterization tools like TEM, XRD, or FTIR for deeper microstructural and chemical analysis.
- Perform environmental durability studies to evaluate resistance to moisture, UV exposure, and temperature variations.
- Develop Finite Element Analysis (FEA) models to simulate mechanical behavior and predict performance under various loading conditions.

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