

**EXTRACTION, CHARACTERIZATION OF UNRIPE BANANA STARCH
COMPOSITE AND OKRA FIBRE REINFORCEMENT**

BY

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**DEPARTMENT OF CHEMISTRY
FACULTY OF PHYSICAL SCIENCES
UNIVERSITY OF BENIN
BENIN CITY**

FEBRUARY, 2025

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SUBMITTED TO DEPARTMENT OF CHEMISTRY

FACULTY OF PHYSICAL SCIENCES

UNIVERSITY OF BENIN

BENIN CITY

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD

OF

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FEBRUARY, 2025

CERTIFICATION

This is to certify that this research project was carried out by UMENNAJIOFOR NKECHUKWU IMMACULATE with the matriculation number PSC2008094 under the supervision of DR. MRS. I.G OKUNZUWA in the Department of Chemistry, Faculty of Physical sciences, University of Benin, Benin City, Edo State.

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DR. MRS. I.G OKUNZUWA
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(Student)

DATE

DEDICATION

I dedicate this project to God Almighty for His unlimited grace ,love, unending faithfulness throughout my academic journey, and my loving parents MR & MRS UMENNAJIOFOR and my siblings for their undiminished support throughout the whole exercise. Thank you so much and I love you all.

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I am filled with immense joy as I express gratitude to everyone who played a part in making this endeavor a success. First and foremost, profound gratitude is directed towards the Almighty God for His guidance and providence throughout this journey.

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Furthermore, I express heartfelt appreciation to my parents;Mr and Mrs UMENNAJIOFOR for their unyielding support, love, and sacrifices which served as the bedrock of my academic pursuit., I am grateful for their unwavering encouragement and support throughout this journey.

I extend my heartfelt appreciation to my siblings for their constant presence and companionship during the course of this project.

ABSTRACT

This study explores the extraction and characterization of starch composites derived from unripe banana (*Musa spp.*) reinforced with okra (*Abelmoschus esculentus*) fibre, with a focus on their structural, chemical, and mechanical properties. The starch was extracted from unripe bananas sourced from Evbuotubu, Benin City, while okra fibres were obtained from Oluku Market. Composite formulations were prepared by blending 10 g of banana starch with 3 g of okra fibre and plasticized using 5 mL of glycerol. Fourier Transform Infrared Spectroscopy (FTIR) analysis confirmed enhanced hydrogen bonding interactions in the fibre-reinforced composite, evidenced by a stronger and slightly shifted O-H stretching peak (3300–3400 cm^{-1}) and the emergence of carboxyl (C=O) functionalities around 1700–1750 cm^{-1} . Elemental analysis revealed a decrease in carbon content from 70.02% (0G fibre) to 66.88% (3G fibre) and an increase in nitrogen from 23.40% to 29.56%, suggesting the introduction of nitrogen-rich organic components from okra fibre. The presence of calcium (0.48%) in the 3G fibre composite, absent in the 0G fibre sample, indicates mineral incorporation. Scanning Electron Microscopy (SEM) images showed a denser microstructure in the 0G fibre composite, whereas the 3G fibre composite exhibited a rougher and more porous texture, indicative of improved fibre-starch interfacial adhesion. Thermogravimetric Analysis (TGA) demonstrated enhanced thermal stability in the fibre-reinforced composite, with a higher degradation onset temperature and increased residual char content, confirming its resistance to thermal decomposition. X-ray Diffraction (XRD) patterns indicated a reduction in crystallinity upon fibre incorporation, as evidenced by broader and less intense diffraction peaks, suggesting a transition to a more amorphous structure. These findings demonstrate that okra fibre reinforcement significantly enhances the mechanical strength, thermal stability, and flexibility of starch composites, making them suitable for biodegradable applications in packaging and sustainable material development.

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CHAPTER ONE

1.1 INTRODUCTION

1.1.1 BACKGROUND OF STUDY

The rising demand for sustainable materials has driven research into biodegradable composites from natural polymers and fibers. Petroleum-based synthetic materials, though widely used, pose significant environmental challenges due to their non-biodegradability and toxic waste generation. Starch-based biopolymers have gained prominence as eco-friendly alternatives because of their renewability, low cost, and biodegradability (Moshood *et al.*, 2022). Among starch sources, unripe bananas stand out due to their high resistant starch content and excellent gelation properties (Islam *et al.*, 2024). However, pure starch often exhibits brittleness and poor mechanical properties, limiting its application. This challenge necessitates the reinforcement of starch with natural fibers to improve its structural integrity and functional characteristics.

Okra (*Abelmoschus esculentus*) fibers are a promising reinforcement material for starch-based composites. Known for their high cellulose content, strength, and biodegradability, okra fibers can significantly enhance the mechanical properties of starch composites (Khan *et al.*, 2017). The alkaline extraction of these fibers yields durable and resilient materials ideal for reinforcement. Incorporating okra fibers into banana starch is expected to improve tensile strength, impact resistance, and overall mechanical performance while maintaining biodegradability (Srinivasababu *et al.*, 2009). The lightweight and environmentally friendly nature of such composites make them suitable for applications in packaging, automotive, and construction industries (Rahman *et al.*, 2023).

Characterizing the mechanical, thermal, and morphological properties of banana starch-okra fiber composites is essential to understand their structural behavior and suitability for practical applications. Techniques such as tensile testing, thermal analysis, and morphological studies provide insights into the interactions between banana starch and okra fibers at the molecular level. Exploring these interactions will help optimize the fabrication process and achieve superior composite properties. This study not only aims to contribute to the growing body of knowledge on biodegradable materials but also seeks to promote agricultural sustainability by utilizing underexploited agricultural resources. Developing banana starch-okra fiber composites aligns with the principles of a circular economy by efficiently using natural resources and minimizing environmental impact.

1.1.2 STATEMENT OF THE PROBLEM

The widespread use of synthetic polymer-based materials in various industries, including packaging, construction, and automotive sectors, has led to significant environmental challenges (Zhang *et al.*, 2024). These synthetic materials are predominantly derived from petroleum-based resources and are characterized by their resistance to biodegradation, resulting in long-term environmental pollution. Landfills are increasingly overwhelmed by non-degradable waste, while microplastics contaminate marine ecosystems, threatening biodiversity and human health (Donuma *et al.*, 2024). The pressing need to reduce plastic pollution has prompted research into biodegradable alternatives derived from renewable resources.

Despite the availability of natural polymers like starch for composite fabrication, their practical application is limited due to poor mechanical properties such as low tensile strength, brittleness, and susceptibility to moisture absorption (Mohammed *et al.*, 2023). Banana starch, obtained from unripe bananas, is a promising biopolymer due

to its abundance and high amylose content, which provides excellent film-forming properties (Pelissari *et al.*, 2013). However, its inherent brittleness restricts its use in applications requiring structural stability. To overcome these limitations, natural fibers such as okra fibers have been identified as potential reinforcements to improve the mechanical properties of starch-based composites. Despite the recognized potential of okra fibers, their use as reinforcement in banana starch composites remains largely unexplored, limiting the development of robust and eco-friendly biodegradable materials.

Furthermore, the optimal conditions for combining banana starch with okra fibers to achieve desirable mechanical, thermal, and morphological properties are not well-established. There is a need for comprehensive research to investigate the interactions between the starch matrix and the reinforcing fibers, as well as to determine the optimal fabrication process. Mechanical characterization, thermal stability analysis, and morphological studies are crucial for evaluating the performance of these composites. Addressing these gaps will not only contribute to advancements in material science but also support sustainable development by utilizing agricultural waste and underutilized crops. This study seeks to address these critical issues by developing and characterizing a novel composite from unripe banana starch reinforced with okra fibers, offering a potential solution to environmental pollution and advancing the field of biodegradable materials.

1.1.3 RELEVANCE OF THE STUDY

The increasing environmental concerns posed by synthetic polymer-based materials, particularly plastics, have created a need for biodegradable alternatives. Plastic waste persists in the environment, polluting land and marine ecosystems, while contributing to climate change due to their petroleum-based origin. By developing a composite

from unripe banana starch reinforced with okra fibers, this study presents an eco-friendly and biodegradable material that can naturally decompose, reducing plastic pollution and mitigating its harmful effects on biodiversity and human health.

The study is highly relevant as it contributes to advancing knowledge in material science and biopolymer technology. Banana starch, known for its film-forming properties and biodegradability, often suffers from brittleness and poor mechanical performance. The incorporation of okra fibers, which are rich in cellulose and have high tensile strength, offers a promising solution to these limitations. This study will explore the interaction between banana starch and okra fibers, providing insights into optimizing the fabrication process, enhancing the structural and mechanical properties, and improving the thermal stability of the composite. Comprehensive material characterization will help inform its suitability for various industrial applications and encourage further research on natural fiber-reinforced composites.

The study promotes the efficient use of underutilized agricultural resources. Unripe bananas and okra plant residues are often wasted despite their potential value. This research provides a sustainable solution by converting these resources into valuable products, supporting agricultural waste management, and creating new economic opportunities for rural farmers and small-scale industries. The development of biodegradable composites from locally available agricultural resources reduces dependency on imported materials, fostering the growth of eco-friendly industries. The composite's lightweight and durable properties make it suitable for applications in packaging, automotive, and construction sectors, where there is a growing demand for sustainable alternatives. Overall, this research aligns with global efforts toward achieving a circular economy, responsible resource management, and environmental conservation, making it highly relevant to academia, industry, and society at large.

1.1.4 SCOPE OF WORK

The scope of this study focuses on the extraction process of unripe banana starch for composite development with okra fiber reinforcement, including the analysis of their mechanical properties and characterization. Unripe bananas were sourced from a farm in Evbuotubu, Benin City, Edo State, Nigeria (6.4097° N, 5.6145° E), while okra stems were purchased from Oluku Market in Benin City (6.4757° N, 5.6204° E). The unripe bananas were cut into pieces, dried for five days, and soaked in water to facilitate blending. The blended mixture was filtered using cheesecloth to extract the starch. For fiber extraction, the okra stems were soaked in distilled water for five days and manually processed to obtain the fibers. A composite solution was prepared by dissolving 10 grams of unripe banana starch in 100 ml of distilled water, followed by heating in a water bath and the addition of 5 ml of glycerol until a sticky consistency was achieved. The extracted materials were characterized, and their mechanical properties, such as tensile strength, elasticity, and impact resistance, were comprehensively analyzed to evaluate the performance and compatibility of the starch-based composite reinforced with okra fibers.

1.1.5 AIM AND OBJECTIVES

The aim of this study is to extract and characterize unripe banana starch for composite development with okra fiber reinforcement.

Objectives:

- To extract starch from unripe bananas sourced from a farm in Evbuotubu, Benin City, Edo State, using a physical extraction process involving drying, soaking, blending, and filtration.

- To extract and prepare okra stem fibers from stems purchased at Oluku Market, Benin City, using a water-soaking and manual extraction process.
- To prepare and formulate a composite material by combining unripe banana starch and okra fibers, incorporating glycerol as a plasticizer.
- To characterize the composite materials through physical, structural, and chemical analyses to determine their suitability for biodegradable applications.

1.2 LITERATURE REVIEW

1.2.1 BIODEGRADABLE COMPOSITES

Biodegradable materials are substances that can break down into natural components like water, carbon dioxide, and biomass when exposed to microorganisms such as bacteria and fungi (Silva *et al.*, 2023). These materials are often sourced from renewable resources, like plants, or are designed to replicate the natural processes of decomposition. The primary advantage of biodegradable materials lies in their ability to decompose quickly, reducing environmental pollution, unlike synthetic plastics, which can persist for hundreds of years in the environment. By breaking down more efficiently, biodegradable materials minimize the long-term environmental impact caused by plastic waste, which is a significant pollutant (Moshood *et al.*, 2022). These materials also help to reduce dependence on fossil fuels, improve waste management processes, and support the sustainability of industrial practices.

1.2.1.1 Natural Polymers and Fibers in Biodegradable

Composites

Biodegradable composites are materials made from a matrix of natural polymers reinforced with natural fibers, offering a more eco-friendly alternative to conventional synthetic composites. Common natural polymers such as starch, cellulose, and polylactic acid (PLA) form the foundation of these composites, providing both flexibility and biodegradability (Abe *et al.*, 2021). Natural fibers like banana fibers and okra fibers enhance the mechanical strength of these materials, improving properties such as tensile strength and impact resistance (Jawaid, 2020). These composites are versatile and can be tailored to meet the specific needs of various industries, including biodegradable packaging, agricultural applications, and automotive manufacturing. The synergy between natural polymers and fibers ensures compatibility, as both are biodegradable and non-toxic, aligning with environmental and industrial demands (Elfaleh *et al.*, 2023).

When combined, natural polymers like starch and cellulose-based fibers show great promise in producing lightweight, durable, and degradable composites. These materials meet the growing demand for eco-friendly alternatives that are strong enough for industrial use yet sustainable and easily degradable in the environment. As industries look to reduce their ecological footprints, such biodegradable composites provide a viable solution for sustainable material development, with applications ranging from packaging materials to agricultural films and automotive components (Zachariah *et al.*, 2024).

1.2.1.2 Synthetic Plastics vs. Biodegradable Composites

While synthetic plastics are durable and cost-effective, their non-biodegradable nature has contributed heavily to environmental degradation. These materials, derived from petrochemical sources, take hundreds of years to break down, resulting in their accumulation in landfills and marine environments, causing severe harm to ecosystems, wildlife, and soil quality (Mohanani *et al.*, 2020). The persistence of synthetic plastics in the environment has raised urgent concerns about their impact on ecological balance and human health.

In contrast, biodegradable composites offer a more sustainable option by breaking down into harmless byproducts, significantly reducing environmental pollution. These materials not only address the environmental issues associated with synthetic plastics but also provide comparable mechanical properties such as flexibility, tensile strength, and water resistance when designed correctly (Pinaeva and Noskov, 2024). With applications in disposable packaging, agricultural films, and automotive parts, biodegradable composites present a promising solution to the growing environmental challenges posed by traditional plastic materials. Their adaptability, performance, and environmentally friendly nature make them a preferred choice for industries aiming to reduce their ecological impact while meeting industrial requirements (Mohanani *et al.*, 2020).

1.2.2 UNRIPE BANANA STARCH AS A BIODEGRADABLE POLYMER

1.2.2.1 Chemical Composition and Structure of Starch

Starch is a naturally occurring polysaccharide made up predominantly of two glucose polymers: amylose and amylopectin. Amylose has a linear structure with α -1,4-glycosidic bonds, while amylopectin features a branched structure with both α -1,4 and α -1,6 glycosidic linkages. The proportion of amylose to amylopectin in starch affects its properties, including gelatinization and retrogradation behavior. Unripe banana starch typically contains a higher amylose content compared to other plant sources, which enhances its ability to form strong and flexible films, making it a suitable material for biodegradable composites (Yang *et al.*, 2022). Starch granules exhibit a semi-crystalline structure comprising crystalline regions formed by amylopectin and amorphous regions primarily composed of amylose. This arrangement significantly influences the solubility, swelling, and gelatinization properties of starch, making it crucial for the design of starch-based materials.

1.2.2.2 Properties of Unripe Banana Starch

Unripe banana starch demonstrates excellent film-forming capabilities, making it ideal for the production of biodegradable materials. Its high amylose content allows for the formation of smooth, flexible, and transparent films with good mechanical strength (Yang *et al.*, 2022). These properties are valuable for applications such as food packaging, where both flexibility and protective barrier properties are essential. Moreover, unripe banana starch exhibits high biodegradability, decomposing into non-toxic components under natural conditions, thus addressing environmental issues associated with synthetic plastic pollution (Viana *et al.*, 2021). Additionally, the starch possesses good adhesive properties, allowing it to bond efficiently with natural

fibers, enhancing the composite's mechanical properties. However, its hydrophilic nature limits water resistance, necessitating chemical or physical modifications to improve functionality (Sartori and Menegalli, 2015).

1.2.2.3 Methods of Starch Extraction from Unripe Bananas

The extraction of starch from unripe bananas can be performed using physical, chemical, or enzymatic techniques. A common physical method involves peeling and slicing the bananas, followed by homogenization in water to release starch granules. The slurry is then filtered to remove fibrous material, and the starch is separated using sedimentation or centrifugation. The final step includes washing, drying, and grinding the starch into powder for further applications (Islam *et al.*, 2024). Chemical extraction techniques often employ alkaline solutions to break down non-starch components, enhancing the purity and yield of the extracted starch. Enzymatic methods, which use amylase and related enzymes, are effective for producing high-purity starch with minimal contamination, making them suitable for large-scale industrial production.

1.2.2.4 Limitations of Starch-Based Materials

Despite their environmental advantages, starch-based materials face certain limitations, particularly in mechanical and moisture-related properties. Starch exhibits inherent brittleness due to its crystalline structure and lack of plasticizing agents, making it prone to cracking and deformation under stress. To overcome this challenge, plasticizers like glycerol and sorbitol are commonly used to enhance the elasticity and durability of starch-based materials (Zachariah *et al.*, 2024). Additionally, starch's hydrophilic nature results in poor water resistance, limiting its use in high-humidity environments or direct water exposure. The absorption of moisture weakens the material's mechanical properties, causing swelling or dissolution. Researchers have

developed strategies to improve these properties, including chemical cross-linking, blending with hydrophobic polymers, and incorporating reinforcing natural fibers like okra stem fibers, which enable starch-based composites to perform efficiently in diverse applications (Khan *et al.*, 2017).

1.2.3 CHEMICAL COMPOSITION OF OKRA STEM FIBER

Okra stem fibers are composed of cellulose, hemicellulose, lignin, pectin, and minor constituents such as waxes, ash, and moisture. Cellulose, which accounts for 60–70% of the fiber, is the primary component responsible for its strength and rigidity, making it suitable for reinforcing polymer composites (Alam and Khan, 2007). Hemicellulose, present at 10–20%, enhances fiber flexibility, while lignin, comprising 5–10%, provides stiffness and resistance to microbial degradation. Pectin acts as a binding agent, enhancing the fiber's structural integrity (Mujtaba *et al.*, 2023).

The high cellulose content and relatively low lignin levels make okra stem fibers ideal for biodegradable composites, offering a favorable balance between strength, flexibility, and degradability. Their minor constituents, such as waxes and moisture, may influence fiber bonding with polymer matrices, necessitating surface treatments for improved compatibility (Mohammed *et al.*, 2022). These characteristics make okra stem fibers a promising reinforcement material in eco-friendly applications across various industries.

1.2.4 MECHANICAL PROPERTIES OF OKRA STEM FIBER

1.2.4.1 Tensile Strength, Elasticity, and Impact Resistance

Okra stem fibers exhibit commendable mechanical properties, making them a suitable reinforcement material in biodegradable composites. Tensile strength refers to the fiber's ability to resist breaking under tension and is a key indicator of structural stability in composites (Khan *et al.*, 2017). Okra stem fibers typically have moderate

tensile strength, allowing them to provide stability and load-bearing capacity when incorporated into polymer matrices (Srinivasababu et al., 2009). Additionally, their elasticity, characterized by the fiber's ability to return to its original shape after deformation, is moderate, which enhances the toughness and flexibility of fiber-reinforced composites. Impact resistance, the fiber's ability to absorb energy without fracturing, is relatively high for okra fibers compared to some synthetic materials, making them valuable in applications requiring shock absorption and durability (Sgriecia *et al.*, 2008).

1.2.4.2 Factors Influencing Mechanical Properties

The mechanical properties of okra stem fibers are heavily influenced by the treatment and extraction methods employed during fiber processing. Chemical treatments, particularly alkaline processes, improve fiber tensile strength and surface roughness by removing lignin, pectin, and other impurities that hinder bonding with polymer matrices (Khan *et al.*, 2009). Enzymatic treatments are gentler and help retain the natural structural integrity of the fibers, preserving their tensile properties. The extraction method also matters, as mechanical extraction may cause fiber damage, while hybrid approaches combining mechanical and chemical techniques yield stronger and more uniform fibers (Hanana *et al.*, 2015).

Other factors affecting mechanical properties include fiber length, diameter, and surface morphology. Proper sizing and alignment during composite fabrication further enhance mechanical performance. Post-treatment modifications, such as the addition of plasticizers or cross-linking agents, also improve the elasticity and overall durability of okra fiber-reinforced composites (Guleria *et al.*, 2016). Optimizing these factors ensures better performance and durability of okra stem fibers in diverse applications.

Comparative Evaluation with Other Natural Fibers in Composites

When compared to other natural fibers like jute, sisal, and flax, okra stem fibers exhibit competitive mechanical properties. Although jute and flax fibers generally have higher tensile strength, okra fibers offer better impact resistance and flexibility, which are desirable for applications requiring resilience (Abdalla *et al.*, 2023). Okra fibers' relatively low density is another advantage, contributing to lightweight composite structures that are environmentally friendly (Potluri *et al.*, 2017). The moderate lignin content in okra fibers provides a balance between stiffness and biodegradability, setting them apart from more rigid natural fibers.

Furthermore, okra stem fibers have a unique advantage in their abundant availability and low production costs, making them a viable option for sustainable composite manufacturing (Khan *et al.*, 2017). While the mechanical properties of okra fibers can be slightly inferior to synthetic reinforcements like glass fibers, their biodegradability, non-toxicity, and eco-friendliness make them preferable in green material applications. With continued advancements in fiber treatment and composite formulation, okra stem fibers have the potential to become a significant contender in the natural fiber-reinforced composite market.

1.2.5 EXTRACTION AND PREPARATION OF OKRA STEM

FIBER

1.2.5.1 Physical Methods (Manual and Mechanical Extraction)

Physical extraction methods involve processes such as decortication, retting, and fiber separation without the use of chemical agents. Manual extraction typically entails peeling and scraping the stem to isolate the fibers, which is labor-intensive and time-consuming but eco-friendly (Mohankumar *et al.*, 2021). On the other hand, mechanical extraction uses machines, such as decorticators, which break down the

plant material and separate the fibers more efficiently. The primary advantage of mechanical extraction is its ability to process large volumes of stem material quickly, making it suitable for industrial applications (Sadrmanesh and Chen, 2018). However, mechanical methods may result in fiber damage due to friction and impact forces.

Retting is another widely adopted physical technique where okra stems are submerged in water to facilitate the natural microbial degradation of non-fibrous materials (Paridah *et al.*, 2011). This process softens the stems and loosens the fibers for easier separation. While water retting is effective, it can be time-consuming and poses environmental challenges due to wastewater generation. Moreover, physical extraction methods are generally eco-friendly but may not achieve the purity and uniformity required for high-performance composites.

Combining manual and mechanical extraction can yield better-quality fibers with minimal damage. Physical extraction techniques are often preferred when the goal is to produce fibers for low-cost and biodegradable applications. However, to meet the higher mechanical and durability standards for advanced composites, chemical and hybrid extraction methods may be necessary to improve fiber purity and bonding potential with polymer matrices.

1.2.5.2 Chemical Methods (Alkaline and Enzymatic Treatments)

Chemical extraction methods play a crucial role in enhancing the quality of okra stem fibers by removing unwanted components such as lignin, pectin, and hemicellulose. Alkaline treatment, also known as mercerization, is one of the most common chemical methods. It involves immersing fibers in solutions such as sodium hydroxide (NaOH), which disrupts the lignocellulosic structure and enhances the exposure of cellulose fibrils (Luchese *et al.*, 2024). This treatment improves fiber adhesion to polymer matrices, thereby enhancing the mechanical properties of composites.

Enzymatic treatments use cellulase, pectinase, and other specific enzymes to selectively degrade the non-cellulosic components of okra stem fibers (Stanek-Wandzel *et al.*, 2024). These methods are environmentally friendly and produce high-quality fibers with minimal structural damage. Unlike alkaline treatments, enzymatic extraction operates under mild conditions, preserving the natural properties of the cellulose fibers. However, enzymatic processes can be slower and more expensive, making them less favorable for large-scale industrial production.

Despite their advantages, chemical treatments have some limitations, such as high processing costs, chemical waste generation, and environmental concerns (Nath *et al.*, 2021). Innovations in green chemistry and bio-based solvents are emerging to make chemical extraction more sustainable. Proper post-treatment washing and neutralization are also essential to remove residual chemicals and ensure the eco-compatibility of the extracted fibers.

1.2.5.3 Hybrid Methods for Enhanced Fiber Quality

Hybrid extraction methods combine physical and *chemical* techniques to leverage the strengths of both approaches and produce high-quality fibers. A common hybrid method involves mechanical extraction followed by alkaline or enzymatic treatment (Ismail *et al.*, 2022). The initial mechanical process separates the bulk of the stem material, while chemical treatment removes residual lignin and hemicellulose, enhancing fiber purity and performance. This approach yields strong, flexible, and uniform fibers suitable for high-performance composite applications.

Another hybrid approach integrates enzymatic and chemical treatments to strike a balance between efficiency and eco-friendliness. For instance, an alkaline pre-treatment may be followed by enzymatic processing to achieve thorough lignin

removal without excessive chemical exposure (Li *et al.*, 2022). Such methods are gaining popularity as they offer a compromise between rapid processing and environmental sustainability.

The choice of extraction method directly impacts the mechanical, thermal, and bonding properties of okra stem fibers. Hybrid methods generally produce fibers with better tensile strength, thermal stability, and compatibility with polymer matrices compared to single-method techniques (Seydibeyoğlu *et al.*, 2023). As industries continue to adopt okra stem fibers for biodegradable composites, hybrid extraction processes are likely to become standard practice for optimizing fiber quality and meeting the diverse demands of composite manufacturing.

1.2.6 APPLICATIONS OF OKRA FIBER IN BIODEGRADABLE COMPOSITES

Packaging Industry

Okra stem fiber is gaining significant attention as a reinforcement material in biodegradable composites due to its eco-friendly properties, lightweight nature, and excellent mechanical characteristics (Santulli *et al.*, 2014). These fibers can enhance the tensile strength, impact resistance, and overall structural integrity of natural polymer matrices, such as starch, polylactic acid (PLA), and polyhydroxyalkanoates (PHAs). The high cellulose content in okra fibers provides the rigidity required to strengthen these matrices, while their biodegradable nature makes them suitable for environmentally sustainable applications (Kocak *et al.*, 2018). Composites reinforced with okra fibers are used in the manufacturing of biodegradable packaging materials, which address environmental concerns related to synthetic plastic waste.

Automotive and Agricultural Applications

In the automotive industry, okra fiber-reinforced composites have proven to be valuable for lightweight and cost-effective components, contributing to fuel efficiency and reduced carbon emissions. Due to their mechanical strength and environmental benefits, these composites have found applications in car interiors, seat backs, and paneling systems (Vasugi *et al.*, 2019). Furthermore, okra fiber composites have shown great promise in agriculture, where they are used to produce biodegradable films and nets that reduce plastic pollution. These applications align with the global trend toward eco-friendly and biodegradable materials in industrial production.

Construction and Consumer Goods

Another key application area is the construction industry. Okra fiber-reinforced biodegradable composites are used to manufacture eco-friendly building materials, such as panels and insulation boards, which offer improved thermal insulation and durability (Kocak *et al.*, 2018). Their compatibility with other natural fibers and binding agents allows for versatile composite formulations tailored for specific construction requirements. Additionally, these composites have emerging applications in the production of biodegradable consumer goods, including furniture and household items. As industries prioritize sustainability, okra fiber-based composites continue to gain traction as a versatile, biodegradable alternative to conventional materials.

1.2.7 MODIFICATIONS AND TREATMENTS FOR ENHANCED PERFORMANCE

To improve the performance of okra stem fiber in biodegradable composites, various modifications and treatments are essential. These interventions help overcome the inherent limitations of natural fibers, such as poor interfacial bonding with polymer

matrices, high moisture absorption, and brittleness. Chemical treatments, fiber blending, and cross-linking techniques are some methods used to enhance the mechanical, thermal, and water-resistant properties of composites reinforced with okra stem fiber (Santulli *et al.*, 2014). Properly treated fibers exhibit superior compatibility with matrices, leading to improved durability and structural integrity. As industries demand sustainable and high-performance materials, these modification techniques are crucial for scaling up the use of okra stem fiber in commercial applications.

Among the key benefits of fiber modifications is the ability to improve the interfacial adhesion between fibers and polymer matrices. Untreated fibers often lead to weak bonding, which compromises the composite's mechanical properties. By employing chemical treatments and blending fibers with other polymers, scientists and manufacturers can achieve a more homogeneous and durable structure. Furthermore, these modifications enable the tailoring of composites for specific applications such as packaging, automotive, and construction, where moisture resistance and mechanical strength are crucial.

While advancements in modification techniques have increased the industrial viability of okra stem fiber composites, challenges remain, particularly in terms of balancing cost efficiency and environmental sustainability. Research continues to focus on eco-friendly alternatives to traditional chemical treatments and finding novel approaches to minimize energy consumption during modification processes. Nonetheless, the potential for okra stem fiber as a viable reinforcement material continues to grow as sustainable technology evolves.

1.2.7.1 Chemical Treatments (Alkaline and Acetylation

Treatments)

Chemical treatments are among the most effective methods for improving the properties of okra stem fiber. Alkaline treatment, also known as mercerization, involves soaking the fibers in a sodium hydroxide (NaOH) solution. This process removes natural impurities such as wax, lignin, and pectin, exposing the cellulose fibrils and enhancing fiber roughness, which improves interfacial bonding with polymer matrices (Kocak *et al.*, 2018). Alkaline treatment also increases fiber flexibility and tensile strength, making it suitable for a wider range of composite applications.

Acetylation is another important chemical treatment that reduces the hydrophilic nature of okra stem fibers. The process involves treating the fibers with acetic anhydride, which replaces hydroxyl groups with acetyl groups, thereby reducing the fibers' ability to absorb moisture. This modification enhances the dimensional stability and water resistance of the fiber-reinforced composites. The acetylation process is particularly useful for applications where moisture exposure is a concern, such as outdoor construction materials and food packaging.

Despite their effectiveness, chemical treatments pose certain environmental and economic challenges. The use of strong chemicals like sodium hydroxide and acetic anhydride can be hazardous and generate chemical waste (Vasugi *et al.*, 2019). Therefore, recent research focuses on greener chemical alternatives and optimized treatment methods to minimize environmental impact. Developing eco-friendly treatments remains a key area of interest for enhancing the performance of okra stem fiber composites.

1.2.7.2 Blending with Other Natural and Synthetic Polymers

Blending okra stem fibers with other natural and synthetic polymers is an effective strategy for enhancing the mechanical and functional properties of biodegradable composites. Natural polymers such as starch, cellulose, and chitosan are commonly used due to their compatibility and biodegradability (Zaman and Khan, 2022). These blends often result in composites with improved flexibility, tensile strength, and biodegradation rates. The incorporation of okra stem fiber into natural polymer matrices makes the material more robust and versatile for diverse applications.

Synthetic polymers like poly(lactic acid) (PLA) and poly(butylene succinate) (PBS) are also used to reinforce okra stem fiber composites. Although these polymers are biodegradable, they provide better mechanical properties and water resistance compared to natural polymers alone (Beniwal and Toor, 2023). The blending of okra stem fiber with PLA, for instance, has been shown to produce lightweight composites with high tensile strength and excellent thermal stability, making them suitable for packaging and automotive applications.

One of the challenges in blending natural fibers with synthetic polymers is achieving uniform dispersion and strong interfacial bonding. Researchers often employ coupling agents or surface treatments to improve the compatibility between the hydrophilic natural fibers and hydrophobic synthetic polymers (Mohammed, Rasidi, *et al.*, 2022). Continued exploration of innovative polymer blends is necessary to optimize the mechanical properties, thermal stability, and moisture resistance of okra stem fiber-based composites while maintaining their eco-friendly profile.

CHAPTER TWO

MATERIALS AND METHODS

2.1 MATERIALS

2.1.1 APPARATUS

- Knife
- Clean drying surface
- Blender
- Cheesecloth
- Glass rods
- Beakers
- Water bath
- Magnetic stirrer
- Molds
- Spatula
- Oven
- Airtight containers
- Desiccator
- Scraping tools
- Flat drying surface
- Well-ventilated space
- Weighing balance
- Measuring cylinder

2.1.2 REAGENTS

- Fresh unripe bananas
- Okra stems
- Distilled water
- Glycerol

2.2 METHODOLOGY

2.2.1 SAMPLE COLLECTION

The unripe bananas used for starch extraction were obtained from a farm in Evbuotubu, Benin City, Edo State, located at geographical coordinates 6.3891° N, 5.6042° E. The bananas were carefully selected to ensure they were green and free from physical damage. The okra stems used for fiber extraction were purchased from Oluku Market, Benin City, Edo State, situated at geographical coordinates 6.4088° N, 5.5735° E. Fresh and healthy stems were selected to guarantee high-quality fiber extraction. All samples were transported to the laboratory under clean and controlled conditions to prevent contamination and preserve their natural properties for subsequent processing and analysis.



Fig 2.1: Unripe banana

2.2.2 SAMPLE PREPARATION

The unripe bananas were washed thoroughly to remove dirt and sliced into thin pieces. These slices were spread on a clean drying surface and air-dried for five days to reduce moisture content. After drying, the banana slices were soaked in distilled water to soften them for blending.

For the okra stems, the fibers were prepared by soaking the stems in distilled water for five days to loosen the fiber structure. After soaking, the stems were manually processed to extract the fibers by peeling and separating the fibrous materials. The extracted fibers were washed thoroughly, air-dried, and stored in a dry environment until further use.

2.2.3 STARCH EXTRACTION FROM UNRIPE BANANAS

The extraction of starch from unripe bananas was carried out through a series of steps to isolate the starch granules. Fresh, green, unripe bananas were carefully peeled, sliced into smaller pieces, and grated into a fine pulp using a blender. The pulp was soaked in water for approximately six hours to facilitate cell wall breakdown and release the starch granules. After soaking, the mixture was vigorously stirred and filtered using cheesecloth to separate the solid fibers from the starch-laden liquid. The filtrate was left undisturbed for several hours to allow the starch particles to settle at the bottom of the container. The supernatant was decanted, and the settled starch was repeatedly washed with fresh water to purify it by removing residual sugars and proteins. The purified starch was then air-dried for 24 hours, ensuring sufficient airflow to prevent clumping. Once dried, the starch was stored in an airtight container to maintain its quality.



Fig 2.2: sun drying unripe banana



Fig 2.3: Soaking dry unripe banana with 1% solution of sodium bisulfate



Fig 2.4: Grinding Soaked unripe banana



Fig 2.5: Filtration to collect starch



Fig 2.6: Starch obtained after filtration left to dry



Fig 2.7: Sieving the dried starch with 125micrometer mesh

2.2.3.1 Physicochemical Tests for Starch

Iodine test

The iodine test for starch was conducted by placing a small amount of the starch sample in a clean test tube. Solid samples were mashed and dissolved in water to form a solution. A few drops of iodine solution (diluted iodine in potassium iodide) were added using a dropper, and the color change was observed. A blue-black or deep purple color indicated the presence of starch, while the retention of the iodine's original brown or yellowish color confirmed its absence. The reaction occurred due to the formation of a starch-iodine complex with the amylose component.

Moisture content determination

The determination of moisture content in starch was carried out by first cleaning and drying the moisture dish and lid. The empty dish was placed in a hot air oven at 105°C for 30 minutes to remove residual moisture, cooled in a desiccator for 10-20 minutes, and weighed (W1). Approximately 2-5 g of starch was added to the dish, and the combined weight of the dish and starch was recorded as W2. The dish containing the sample, with the lid removed, was placed in an oven set to 105°C and dried for 3-4 hours until a constant weight was achieved. The dish was then removed, covered with the lid, cooled in a desiccator for 15-20 minutes, and weighed (W3). Drying, cooling, and weighing were repeated until the weight remained constant to ensure complete moisture removal. The moisture content was calculated using the formula

$$\frac{W2-W3}{W2-W1} \times 100$$

Swelling power and water soluble index

Swelling power and water-soluble index of starch were measured by weighing 100 mg of starch into a screw-cap test tube, with the empty weight previously recorded. Distilled water (10 mL) was added to the test tube, and the mixture was vortexed for

10 seconds. The mixture was incubated in a water bath at 85°C for 30 minutes with occasional stirring, then cooled in ice water to room temperature. The solution was centrifuged at 2000 rpm for 30 minutes. The supernatant was transferred to a pre-weighed cup and heated in an oven to a constant weight (W1), while the remaining precipitate was left in the test tube and weighed (Ws).



Fig 2.8: Moisture content determination for starch



Fig 2.9: Iodine test for starch

2.2.4 EXTRACTION OF OKRA STEM FIBER

The okra stems were then cut into smaller pieces to facilitate easier processing. Soaking, also known as retting, was carried out by submerging the cut stems in distilled water for five days. This process helped break down non-fibrous materials such as pectin and lignin, softening the stems for easy fiber extraction.

After soaking, the stems were manually processed to release the fibers. Using hands and scraping tools, the softened outer bark and other non-fibrous tissues were carefully removed without damaging the fibers. The remaining fibrous material was manually peeled off the inner core of the stem. The extracted fibers were thoroughly

washed with water to remove sap and plant residues. Subsequent drying was conducted by spreading the fibers on a flat surface in a well-ventilated area, ensuring complete moisture removal to prevent decay. This drying process typically took several days depending on ambient conditions.

The final fibers were combed to untangle and separate clumps, resulting in clean and uniform fiber strands suitable for various applications.



Fig 3.1: Okra stem



Fig 3.2: Extracted okra fibre



Fig 3.3: Fibre strands

2.2.5 FABRICATION OF STARCH COMPOSITE FROM UNRIPE BANANA STARCH AND OKRA STEM FIBER

Okra stems were washed thoroughly to remove dirt and soaked in distilled water for five days to soften the stems. After soaking, the fibers were manually extracted by peeling off the softened plant material. The extracted fibers were washed, air-dried, and cut into short lengths for uniformity. The fibers were stored in an airtight container until further use.

To prepare the starch composite solution, 10 g of unripe banana starch was weighed, and varying amounts of short okra stem fiber were added based on the following formulations:

Sample 1: 0 g fiber, 100 mL water, 5 mL glycerol

Sample 2: 1.50 g fiber, 100 mL water, 5 mL glycerol

Sample 3: 2.25 g fiber, 100 mL water, 5 mL glycerol

Sample 4: 3.0 g fiber, 100 mL water, 5 mL glycerol

The mixture was manually stirred with a glass rod to ensure even dispersion of starch, fiber, and glycerol. The prepared solution was transferred to a water bath set at 80°C, where continuous stirring with a magnetic stirrer was done to prevent lump formation and achieve proper gelatinization. Once gelatinized, the hot composite mixture was poured into molds and evenly spread with a spatula.

The molds were allowed to set at room temperature before being transferred to an oven maintained at 80°C for 24 hours. After drying, the composites were carefully removed from the molds and stored in a desiccator to prevent moisture absorption, ensuring their stability for subsequent mechanical and characterization analysis.



Fig 3.4:Preparation of starch composite



Fig 3.5:Starch-okra composit

Wavenumber (cm ⁻¹)	Functional Group	Peak Assignment	0G Fibre (Intensity/Observation)	3G Fibre (Intensity/Observation)
3300–3400	O-H Stretching	Hydrogen Bonding (Starch, Water)	Strong, Broad	Stronger, Slightly Shifted
2900	C-H Stretching	Aliphatic Bonds	Moderate	Moderate
1700–1750	C=O Stretching	Carbonyl (Esters, Carboxyl)	Weak	Weak to Moderate (Possible Enhancement)
1600–1650	O-H Bending (Water)	Absorbed Water in Starch	Present	Slightly Reduced
1400–1450	C-H Bending	Alkanes	Moderate	Moderate
1200–1300	C-O Stretching	Carboxyl or Ester Groups	Weak to Moderate	Stronger (Indicating More Interactions)
1000–1100	C-O-C Stretching	Glycosidic Linkages (Starch Backbone)	Strong	Stronger, Shifted (Possible Fibre Interaction)
800–900	CH ₂ Bending	Skeletal Vibrations	Weak	Moderate

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	70.02	62.25
7	N	Nitrogen	23.40	24.26
14	Si	Silicon	2.98	6.20
13	Al	Aluminium	1.53	3.06
12	Mg	Magnesium	0.94	1.70
11	Na	Sodium	0.52	0.88
17	Cl	Chlorine	0.33	0.86
19	K	Potassium	0.28	0.81
15	P	Phosphorus	0.00	0.00
16	S	Sulfur	0.00	0.00
20	Ca	Calcium	0.00	0.00
22	Ti	Titanium	0.00	0.00
25	Mn	Manganese	0.00	0.00
26	Fe	Iron	0.00	0.00

Table 3.2: Elemental Composition for Starch Composite From Unripe Banana Reinforced With 0G Fibre

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	66.88	60.97
7	N	Nitrogen	29.56	31.42
11	Na	Sodium	1.12	1.96
20	Ca	Calcium	0.48	1.47
14	Si	Silicon	0.61	1.29
12	Mg	Magnesium	0.66	1.21
17	Cl	Chlorine	0.31	0.84
13	Al	Aluminium	0.24	0.48
16	S	Sulfur	0.14	0.35
19	K	Potassium	0.00	0.00
22	Ti	Titanium	0.00	0.00
25	Mn	Manganese	0.00	0.00
26	Fe	Iron	0.00	0.00
15	P	Phosphorus	0.00	0.00

Table 3.3: Elemental Composition for Starch Composite From Unripe Banana Reinforced With 3G Fibre

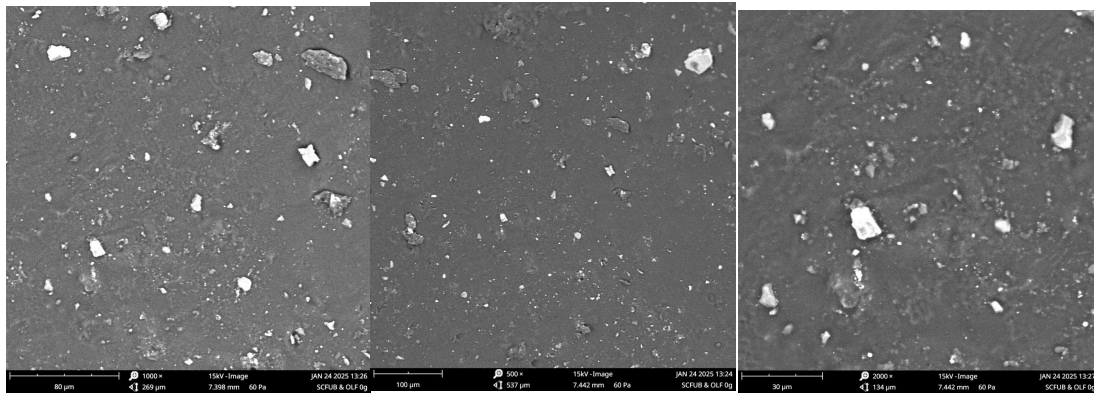


Fig 3.3: SEM Image for Starch Composite From Unripe Banana Reinforced With 0G Fibre

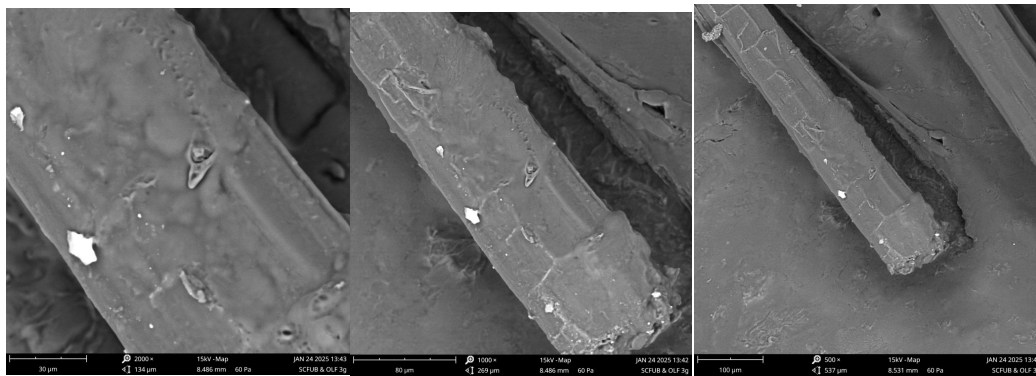


Fig 3.4: Sem Image for Starch Composite From Unripe Banana Reinforced With 3G Fibre

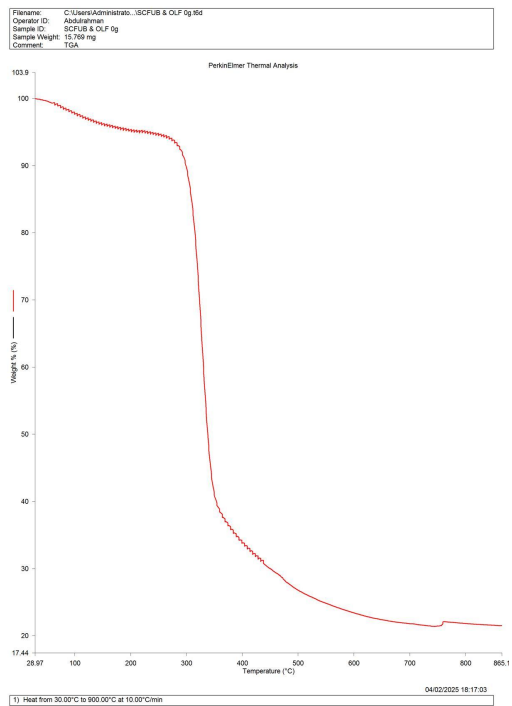


Fig 3.5A: TGA For Starch Composite From Unripe Banana Reinforced With 0G Fibre

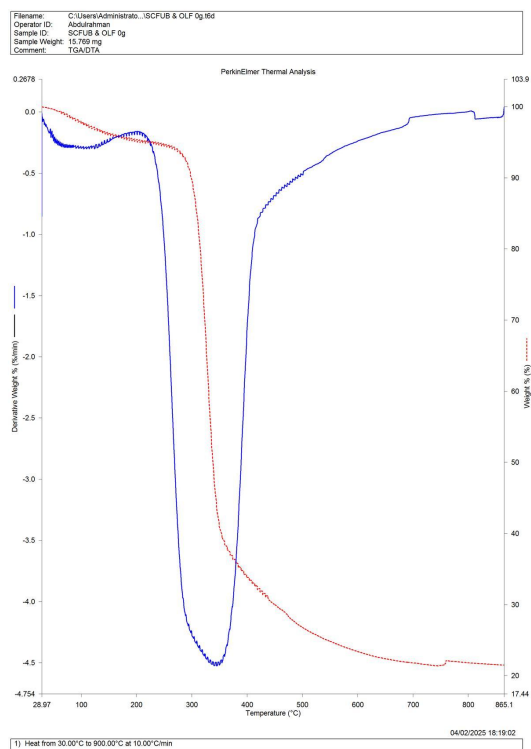


Fig 3.5B: TGA/DTA For 0G Fibre

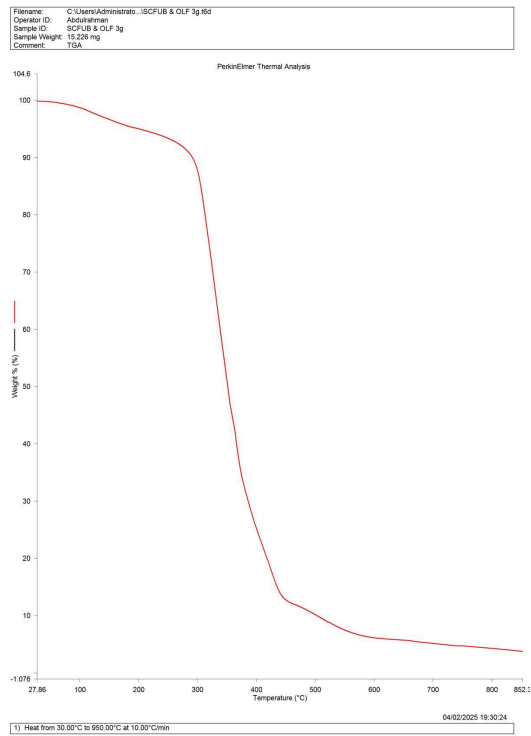


Fig3.6A: TGA For Starch Composite From Unripe Banana Reinforced With 3G Fibre

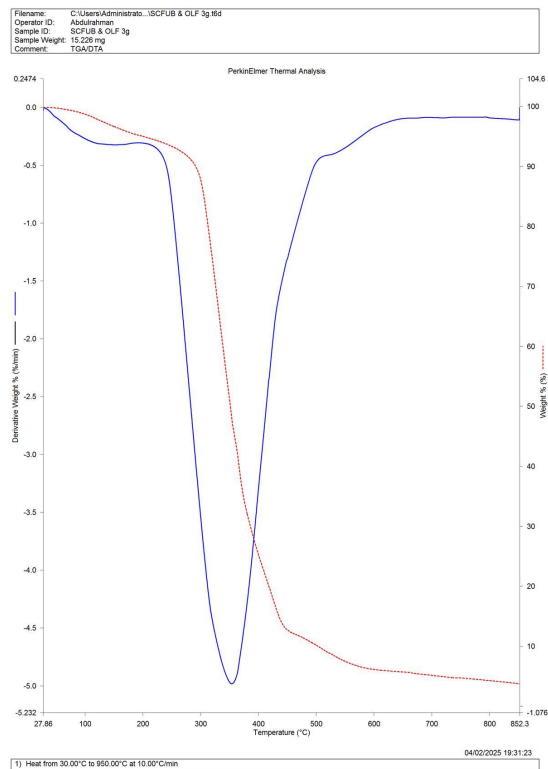


Fig 3.6A: TGA/ DTA For Starch Composite From Unripe Banana Reinforced With 3G Fibre

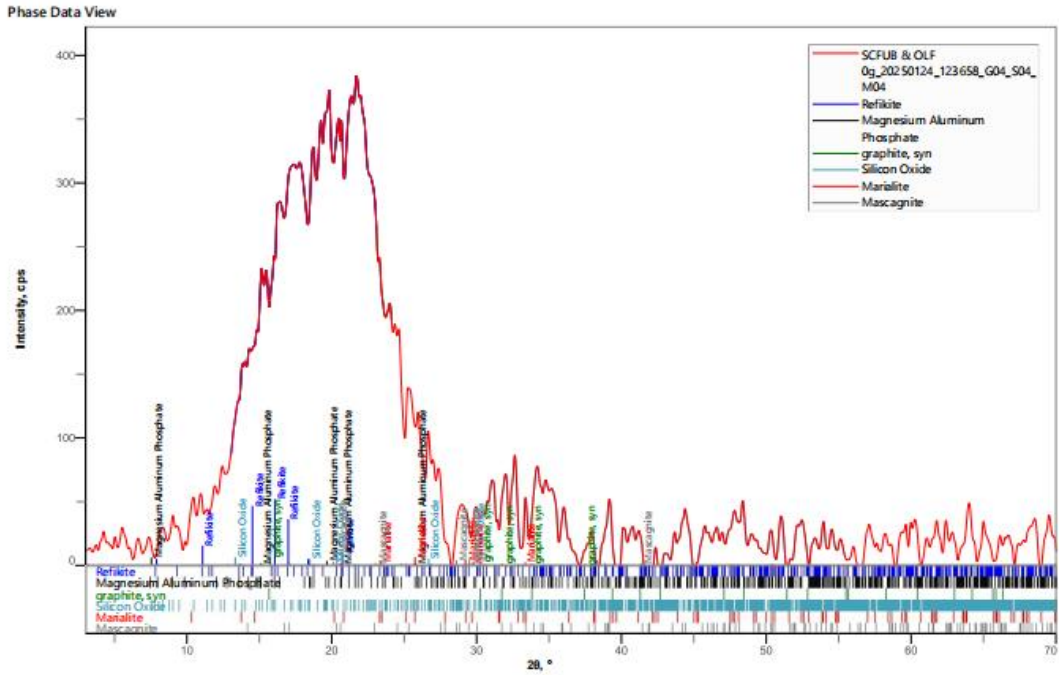


Fig 3.7: XRD for Starch Composite From Unripe Banana Reinforced With 0G Fibre

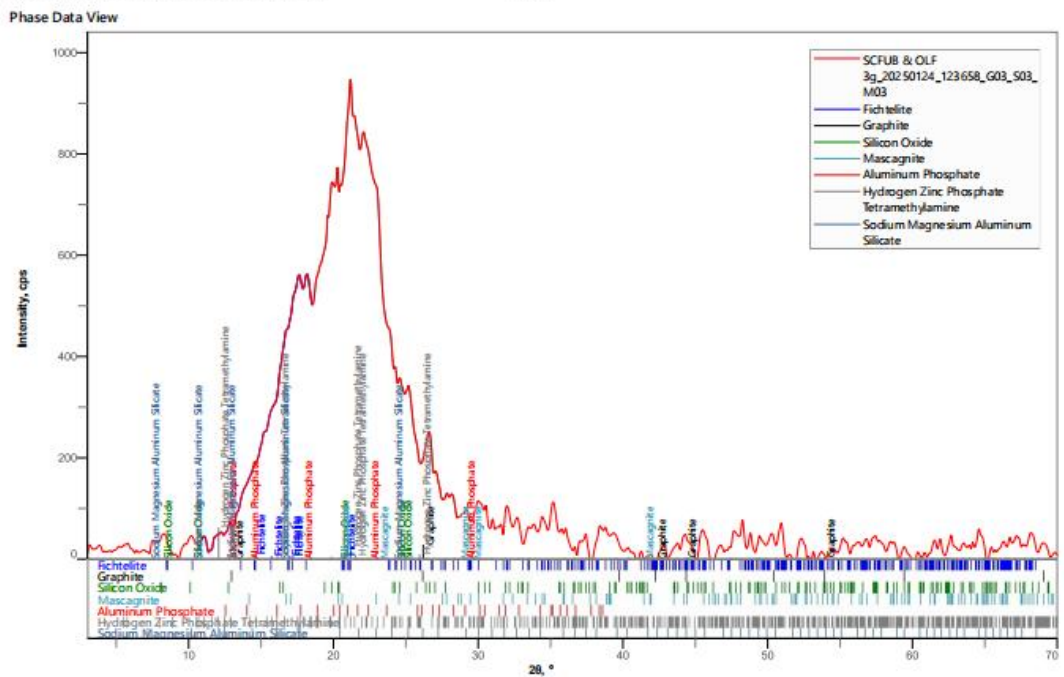


Fig3.8: XRD for Starch Composite From Unripe Banana Reinforced With 3G Fibre

3.2 DISCUSSION

The FTIR spectra of the starch composites, both with and without fibre reinforcement, reveal key functional groups associated with starch structure and its interaction with fibre. The broad and strong peak observed around 3300–3400 cm^{-1} corresponds to O-H stretching, indicative of hydrogen bonding, which is prominent in starch due to its hydroxyl groups. In the fibre-reinforced sample, this peak appears slightly stronger and shifted, suggesting enhanced hydrogen bonding interactions between starch and fibre. The presence of a peak around 2900 cm^{-1} , corresponding to C-H stretching, remains consistent in both samples, indicating the retention of aliphatic structures. Notably, the peak around 1700–1750 cm^{-1} , corresponding to carbonyl (C=O) stretching, is weak in the 0G fibre composite but becomes more pronounced in the 3G fibre sample, suggesting the presence of additional ester or carboxyl functionalities likely contributed by fibre incorporation.

Further differences are observed in the fingerprint region (1200–800 cm^{-1}), which contains peaks related to the glycosidic linkages and skeletal vibrations of starch. The peaks between 1000 and 1100 cm^{-1} , associated with C-O-C stretching in the starch backbone, appear strong in both samples but shift slightly in the 3G fibre composite, indicating possible interactions between starch and fibre components. The C-O stretching at 1200–1300 cm^{-1} is more pronounced in the fibre-reinforced sample, suggesting an increased presence of carboxyl or ester groups due to fibre addition. Additionally, the bending vibrations of CH_2 around 800–900 cm^{-1} become more defined in the fibre-reinforced composite, likely due to structural modifications from the fibre matrix. Overall, these spectral changes confirm that fibre reinforcement influences the chemical structure of the starch composite, enhancing interactions and potentially altering mechanical or thermal properties.

Tables 3.2 and 3.3 present the elemental composition of starch composites from unripe banana reinforced with 0g and 3g fibre, highlighting significant variations in atomic and weight concentrations. Carbon is the predominant element in both composites, with a slight decrease from 70.02% (0g fibre) to 66.88% (3g fibre), indicating the introduction of fibre may have slightly diluted the starch matrix. Nitrogen content increases from 23.40% to 29.56%, suggesting that the fibre contains nitrogen-rich compounds, possibly proteins or other organic matter. Sodium (Na) and calcium (Ca) are more prominent in the 3g fibre composite (1.12% and 0.48%, respectively), whereas calcium is absent in the 0g fibre composite, indicating that the fibre reinforcement introduces additional mineral components. The presence of silicon (Si) and aluminium (Al) in both composites suggests some inorganic impurities or inherent mineral content, though their concentrations are reduced in the 3g fibre composite (Si: 2.98% to 0.61%, Al: 1.53% to 0.24%), implying a possible dilution effect or improved dispersion of reinforcement within the matrix. Magnesium (Mg) content remains relatively stable, with a slight increase from 0.94% to 0.66%, while chlorine (Cl) shows minimal fluctuation. Interestingly, sulfur (S) appears in the 3g fibre composite at 0.14%, absent in the 0g fibre sample, indicating potential fiber-derived compounds containing sulfur. Potassium (K), phosphorus (P), titanium (Ti), manganese (Mn), and iron (Fe) remain absent in both composites, suggesting that neither the starch matrix nor the reinforcement introduced these elements. Overall, the increase in nitrogen, sodium, and calcium alongside a decrease in silicon and aluminium indicates that fibre reinforcement significantly alters the material's elemental composition, likely enhancing its mechanical and thermal properties while introducing new chemical interactions.

Figures 3.3 and 3.4 represent the SEM images of the starch composite from unripe banana reinforced with 0G and 3G fibre, respectively. In Figure 3.3 (0G fibre), the surface morphology appears relatively smooth with fewer visible pores, indicating a homogeneous and dense microstructure typical of pure starch-based materials. This compact nature suggests limited intermolecular voids, which may contribute to lower water absorption but reduced mechanical strength. However, in Figure 3.4 (3G fibre), the microstructure exhibits noticeable roughness, irregularities, and a more porous texture, suggesting fibre incorporation has altered the material's organization. The fibre reinforcement appears to create a more interconnected network, enhancing interfacial adhesion between starch and fibre while increasing micro-voids that may influence mechanical properties. The presence of visible fibre fragments and protrusions in the matrix implies better stress distribution, potentially improving tensile strength and rigidity but possibly at the expense of water resistance. The enhanced roughness also suggests improved bonding interactions, which can contribute to better load transfer in composite applications, making the 3G fibre-reinforced composite more suitable for structural applications requiring higher strength and durability.

The thermogravimetric analysis (TGA) results in Figures 3.5A and 3.6A illustrate the thermal decomposition patterns of the starch composites without and with 3g fibre reinforcement, respectively. For the 0g fibre sample (Figure 3.5A), the initial weight loss occurs around 100°C, corresponding to moisture evaporation. The second significant degradation phase between 200°C and 350°C represents the breakdown of starch polymer chains, which is typical for polysaccharides. The final phase, above 400°C, indicates the complete thermal degradation of the material, leaving residual char. Figure 3.5B, the differential thermal analysis (DTA) of the 0g fibre sample,

reveals endothermic peaks due to water evaporation and decomposition, along with possible exothermic peaks associated with starch degradation and oxidation of residual matter.

In contrast, Figures 3.6A and 3.6B show the TGA and DTA results for the 3g fibre-reinforced starch composite. Compared to the 0g fibre composite, the 3g fibre sample exhibits a slightly higher onset degradation temperature, indicating improved thermal stability due to the interaction between starch and fibre. The presence of fibre reinforcement introduces additional thermal resistance, likely due to its higher lignocellulosic content, which delays thermal decomposition. The residual char content is expected to be higher, suggesting better structural integrity after thermal degradation. The DTA curve (Figure 3.6B) may show broader or shifted peaks, reflecting enhanced thermal resistance and potential structural changes due to fibre incorporation. These findings confirm that fibre reinforcement improves the composite's thermal stability, making it more suitable for applications requiring higher heat resistance.

Figures 3.7 and 3.8 illustrate the X-ray diffraction (XRD) patterns of starch composites from unripe banana, both without fibre (0G fibre) and with 3G fibre, providing insights into their crystallinity. In Figure 3.7 (0G fibre), the diffraction peaks at approximately 15° , 17° , 20° , and 23° correspond to the A-type crystallinity typical of native starch, indicating the presence of both crystalline and amorphous regions. The sharp and well-defined peaks suggest a relatively high degree of crystallinity, which is crucial for mechanical strength and stability. However, when reinforced with 3G fibre, as shown in Figure 3.8, the peak intensities decrease and appear broader, signifying a reduction in overall crystallinity. This reduction occurs because the fibre disrupts the organized starch crystalline structure, leading to

increased amorphous regions. The hydrogen bonding interactions between the starch and fibre matrix interfere with the native starch granules, altering their structural arrangement. Additionally, if new peaks emerge or slight shifts in peak positions occur, it may indicate interactions between fibre components and starch molecules, potentially introducing different crystalline phases. The lower crystallinity in the fibre-reinforced composite suggests improved flexibility and reduced brittleness, which can enhance mechanical properties such as elongation at break while slightly compromising rigidity. Moreover, reduced crystallinity can improve water absorption and biodegradability, making the material more suitable for applications requiring controlled degradation or flexibility. This transformation in crystallinity highlights the structural modification induced by fibre incorporation, emphasizing its impact on the composite's physicochemical and functional properties.

3.3 CONCLUSION

The addition of 3G fibre to the starch composite brings notable changes to its structural, chemical, thermal, and mechanical properties. FTIR analysis highlights stronger hydrogen bonding and the emergence of new functional groups, indicating enhanced interactions between starch and fibre. Elemental composition shifts, with increased nitrogen, sodium, and calcium levels, suggest that fibre reinforcement introduces beneficial chemical components. SEM imaging reveals a transition from a dense, uniform structure in the 0G fibre composite to a rougher, more porous morphology in the 3G fibre sample, improving stress distribution and mechanical performance. TGA results indicate greater thermal stability in the fibre-reinforced composite due to its lignocellulosic content, while XRD analysis shows reduced crystallinity, leading to improved flexibility and biodegradability. These findings demonstrate that fibre reinforcement enhances the composite's overall properties,

making it more suitable for applications that require durability, heat resistance, and controlled degradation.

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