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Thermal, Morphological, and Structural Properties of Biodegradable Unripe Banana Starch (Musa sapientum L.) Composites Reinforced with Pineapple Leaf Fibres, (*Ananas comosus L. Merr.*)

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Abstract

Growing interest in the creation of biodegradable composites is a result of the growing demand for sustainable substitutes for synthetic polymers. The main aim of this study is to characterize and contrast biodegradable composites composed of unripe banana starch reinforced with pineapple leaf fiber. Two composite formulations were created: Sample B (10 g of unripe banana starch, 5 mL of glycerol, 100 mL of distilled water, and 5 g of fiber) and Sample A (10 g) of unripe banana starch, 5 mL of glycerol, 100 mL of distilled water, and no added fiber). Strong intermolecular interactions were confirmed by Fourier Transform Infrared Spectroscopy (FTIR) analysis, as evidenced by the carbonyl (C=O) and hydroxyl (-OH) absorption peaks present in both samples. Significantly, Sample B showed more intense peaks, which suggested that the fiber and starch interacted better and had a stronger hydrogen bond. Surface structure variations were clearly visible using scanning electron microscopy (SEM). While Sample B displayed better fiber dispersion, stronger adhesion between the fiber and starch matrix, with a more uniform texture (signs of improved stress distribution). Sample A had a rough, brittle appearance with visible microcracks. Changes in the material's elemental composition were verified by Energy Dispersive X-ray Spectroscopy (EDX). The fiber was successfully incorporated into the matrix, as evidenced by Sample B's higher carbon content. Sample B had higher crystallinity, according to X-ray diffraction (XRD) analysis, suggesting that fiber reinforcement had improved molecular alignment and increased structural integrity. Overall, the results suggest that pineapple leaf fibre significantly improves the structural, morphological, and mechanical characteristics of the composite. These

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enhancements support its potential as a sustainable and eco-friendly alternative to conventional polymers. With better fibre-matrix bonding, improved molecular structure, and greater mechanical stability, these biodegradable composites hold promise for use in areas such as sustainable packaging, lightweight construction, and environmentally responsible consumer products.

Keywords: *Biodegradable composites, Characterization, Unripe banana starch, Pineapple leaf fibre, Reinforcement.*

1. INTRODUCTION

The global search for sustainable alternatives, particularly in the field of biodegradable materials, has accelerated due to the growing environmental concerns surrounding synthetic polymers (Moshood *et al*., 2022). The durability of synthetic plastics, which are mostly made from petrochemical sources, is well-known, but it also helps explain why they persist in the environment for a long time. Their extensive use has had detrimental ecological effects and caused significant pollution, especially in packaging (Gilani *et al.,* 2023). Biodegradable composites are becoming more and more popular as a solution to these urgent problems. Because they are readily available, renewable, and have a small environmental impact, natural polymers and fibers are particularly appealing (Bhat *et al*., 2021).

Unripe banana starch, a carbohydrate-based polymer, stands out as a promising material for developing biodegradable composites. This is largely thanks to its excellent film-forming ability, flexibility, and natural compatibility with plant-based fibres. (Dilkushi *et al*., 2024). The starch itself is mainly composed of amylose and amylopectin, with amylose being especially important for strengthening and stabilizing bioplastic materials (Abe *et al*., 2021). However, starch-based materials often struggle with brittleness and moisture sensitivity, which limits their practical use. These challenges can be overcome by reinforcing the starch with natural fibres, thereby improving its strength and overall durability (Jayarathna *et al*., 2022).

Among natural fibres, pineapple leaf fibres (PALF) sourced from agricultural waste have gained recognition for their excellent mechanical properties. Known for their high tensile strength and low density, PALF serves as an effective reinforcing agent in composite materials (Sethupathi *et al*., 2024). With their rich content of cellulose, hemicellulose, and lignin, these fibres significantly enhance the tensile and flexural strength of the composites they are added to (Sethupathi *et al*., 2024). When integrated with unripe banana starch, PALF contributes to a synergistic improvement in both mechanical performance and biodegradability (Ishara *et al*., 2024)

This study explores the development and detailed characterization of biodegradable composites reinforced with pineapple leaf fibres, with the goal of meeting the increasing demand for sustainable materials across different industries. The findings offer valuable contributions to the broader effort of reducing plastic pollution by presenting a greener, more environmentally responsible alternative to traditional synthetic polymers.

2. MATERIALS AND METHOD

The main raw materials used in this study were unripe bananas (Musa sapientum L.) and pineapple leaves (Ananascomosus L. Merr.), both were identified by Prof. Akinnibosun Henry Adewale, Department of Plant Biology and Biotechnology, University of Benin, Edo State with voucher numbers UBH-P406 and UBH-A302 respectively. Pineapple leaves were collected from Ohonre Community in Benin City, Edo State, Nigeria (coordinates: 6.1902° N, 5.6097° E), while the unripe bananas were sourced from the Evbuotubu quarters of the same city (coordinates: 6.4016° N, 5.6091° E). All other chemicals includes 1% sodium bisulfite solution, glycerol (used as a plasticizer), 3% sodium hydroxide solution, and distilled water were of analytical grade.

2.1 Extraction of Unripe Banana Starch

Unripe banana starch was extracted following a modified procedure based on the method outlined by Islam *et al.* (2024). The unripe bananas were manually peeled and uniformly sliced using sanitized equipment, including a clean knife and cutting board. The banana slices were subsequently sun-dried on hygienic trays for a duration of one week to significantly reduce their moisture content. Upon completion of drying, the slices were washed with a 1% sodium bisulfite solution to suppress microbial growth and enhance starch recovery. The treated material was homogenized with distilled water using a blender to produce a consistent slurry, which was then filtered through a double layer of cheesecloth to separate fibrous residues. The resulting filtrate was left undisturbed to facilitate sedimentation of the starch. Once the starch had settled, the supernatant was decanted, and the wet starch sediment was collected. This sediment was air-dried over a period of three days, manually ground with a mortar and pestle, and sieved through a 125 μm mesh to obtain a uniform particle size. The final starch product was stored in airtight containers to preserve its quality and prevent moisture absorption.

2.2 Extraction of Pineapple Leaf Fibre

The extraction of pineapple leaf fibre was carried out using the method proposed by Peng *et al*. (2023) with slight modifications. After being cleaned and trimmed, fresh pineapple leaves were cut into 50–70 cm lengths. After that, these were placed in a container with clean water and weighed to make sure they were completely submerged. Lignin and pectin were broken down by natural fermentation during the seven-day retting process. Every day, the water was checked and replaced if there were any unpleasant smells or excessive foaming.Following fermentation, the softened leaves were cut off, and non-fibrous materials were scraped off with a knife to manually extract the fibers. To further purify the extracted fibers by eliminating any remaining plant material, they were soaked in a 3% sodium hydroxide solution for half an hour. To get rid of any last traces of alkali, they were then thoroughly rinsed with distilled water. To guarantee thorough drying, the cleaned fibers were first allowed to air dry for two days before being oven-dried at 40°C.

2.3 Fabrication of Starch Composite from Unripe Banana Starch and Pineapple Leaf Fibre

The fabrication process was based on the method described by Selamat *et al.* (2018), with slight modifications. Pineapple leaf fibres were thoroughly washed to remove impurities, air-dried under ambient conditions, and subsequently cut into short lengths using a mechanical blender. The processed fibres were then sieved to ensure uniform particle size and stored in airtight containers to prevent moisture uptake and contamination. Two distinct composite formulations were subsequently developed:

SAMPLE A	SAMPLE B
0 g fibre	5 g fibre
100 mL water	100 mL water
5 mL glycerol,	5 mL glycerol,
10 g unripe banana starch	10 g unripe banana starch

Each formulation was manually homogenized using a glass rod to ensure uniform distribution of starch, fibre, and glycerol components. The blended mixture was subsequently heated in a water bath maintained at 80 °C, with continuous agitation using a magnetic stirrer to facilitate complete starch gelatinization and to prevent clumping. Upon achieving a homogeneous hot composite solution, the mixture was carefully poured into moulds and evenly spread with a spatula to ensure consistent thickness. After initial setting at ambient temperature, the moulds were transferred to a hot air oven and dried at 80 °C for 24 hours. The resulting biocomposite films were then stored in a desiccator to inhibit moisture absorption prior to subsequent characterization and testing.

2.4 Characterization of Starch Composites

2.5 FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) was carried out using the KBr pellet method and recorded over the 650–4000 cm^{-1} range with a Cary 630 FTIR spectrometer from Agilent Technologies Inc., USA. This analysis was used to identify functional groups, chemical bonds, and the nature of interactions between the starch and pineapple leaf fibre within the composite matrix.

2.6 SEM-EDX Analysis

The surface morphology and elemental composition of the composite samples were analyzed using a scanning electron microscope coupled with energy-dispersive X-ray spectroscopy (SEM-EDX), specifically the Phenom ProX model (Phenom World, Eindhoven, Netherlands). The SEM was employed to examine the topography, microstructural features, and fibre distribution within the biocomposites, while the EDX system facilitated elemental analysis, identifying both major and trace elements present in the samples. These combined analyses were instrumental in assessing the structural uniformity, overall material quality, and potential performance characteristics of the fabricated biocomposites.

2.7 Thermogravimetric Analysis (TGA)

TGA was conducted using the PerkinElmer TGA 4000 (Netherlands) to assess the thermal stability and decomposition behaviour of the composites. This test provided insights into the materials' resistance to heat and the efficiency of the composite's formulation and reinforcement.

2.8 X-ray Diffraction (XRD)

XRD analysis was employed to examine the crystalline structure of the composites. This technique helped identify the types and relative proportions of crystalline phases present in the materials, offering an understanding of how fibre incorporation affects crystallinity and structural integrity.

3. RESULT AND DISCUSSION

3.1 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectroscopy results comfirms the formation of Unripe banana starch **(Musa sapientum** L.) and Unripe banana starch **(Musa sapientum** L.) composites reinforced with Pineapple Leaf Fibres, (*Ananas comosus L. Merr*.). The various confirmation bands are presented in Figure 1 and Figure 2 respectively.

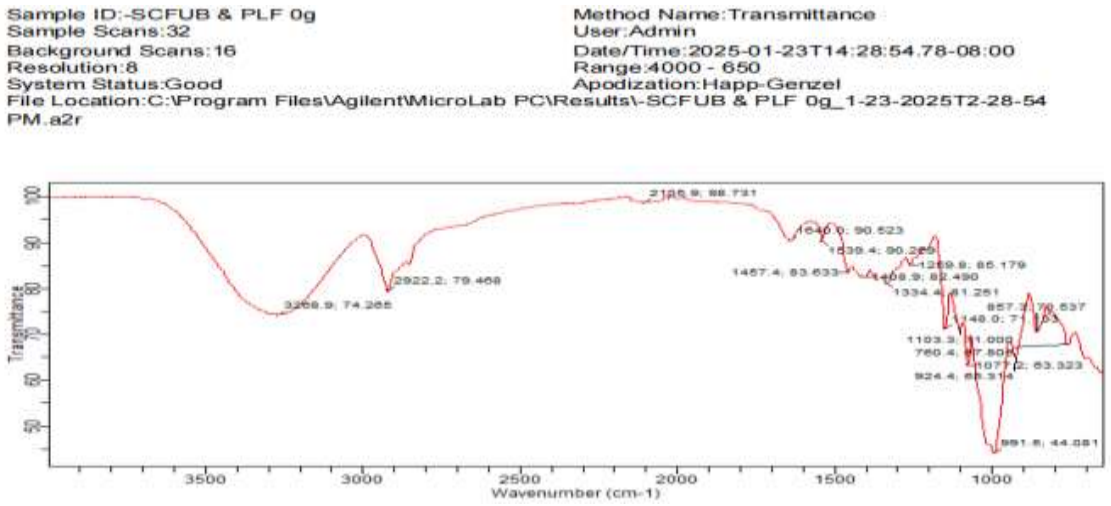

Figure 1: *FTIR for starch composite from 10 g of unripe banana starch containing 0 g of pineapple leaf fibre*

Key functional groups that are characteristic of starch were confirmed by the FTIR analysis of the starch composite made from 10 g of unripe banana starch (0 g fiber). According to Hansen and Spanget-Larsen (2017), hydrogen bonding, which is necessary for water absorption and gelatinization, is indicated by the broad peak at 3268.9 cm^{-1} (O–H stretching). The aliphatic structure of starch molecules is represented by the peak at 2922.2 cm^{-1} (C–H stretching). While 1077.2 cm^{-1} (C–O stretching) validates glycosidic bonds and maintains the starch structure, the 1640.0 cm^{-1} peak (H–O–H bending) indicates the presence of bound water. The aliphatic nature of the starch is further supported by the 760.4 cm^{-1} peak (C–H stretching) (Kumar, *et al.*, 2018). These findings support the starch's structural integrity, though water interactions may have an impact on how well it works in biodegradable applications.

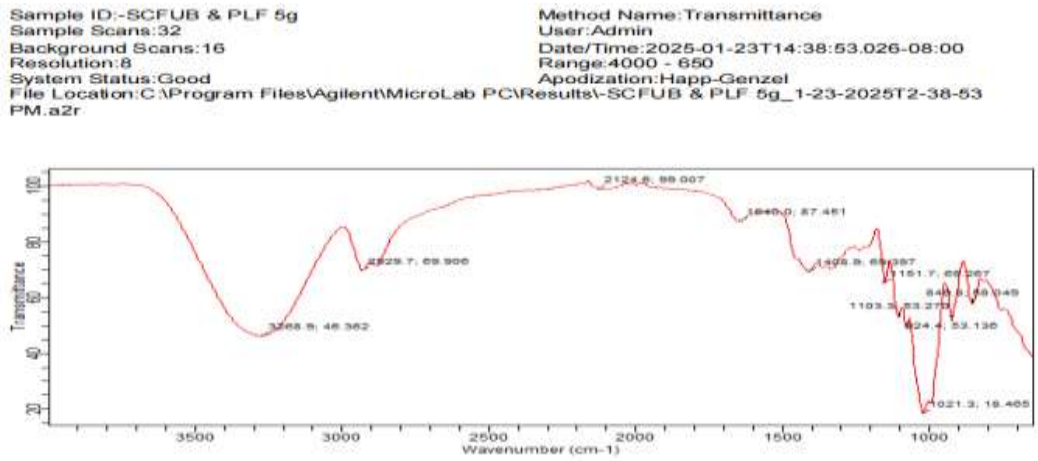

Figure 2: *FTIR for starch composite from 10 g of unripe banana starch containing 5 g of pineapple leaf fibre*

The FTIR analysis of the starch composite with 5 g of pineapple leaf fibre confirmed key functional groups indicating strong fibre-matrix interactions. The O–H stretching at 3268.9 cm^{-1} showed reduced intensity (**46.382**), suggesting enhanced hydrogen bonding between starch and fibre. The C–H stretching at 2929.7 cm^{-1} **(**69.908) and C–H bending at 1408.9 cm^{-1} **(**69.397) confirm the presence of cellulose and lignin from the fibre. The H–O–H bending at 1640.0 cm^{-1} **(**87.451**)** suggests increased water retention, while the C–O stretching at 1021.3 cm^{-1} **(**53.136**)** reflects the glycosidic bonds of starch and fibre. These results confirm improved interfacial interactions, enhancing the composite's structural properties.

3.2 Scanning Electron Microscopy (SEM)

SEM images used for providing insights into the unripe banana starch **(Musa sapientum** L.) material's formation process, degree of homogeneity, and the nature of interactions between its constituents.

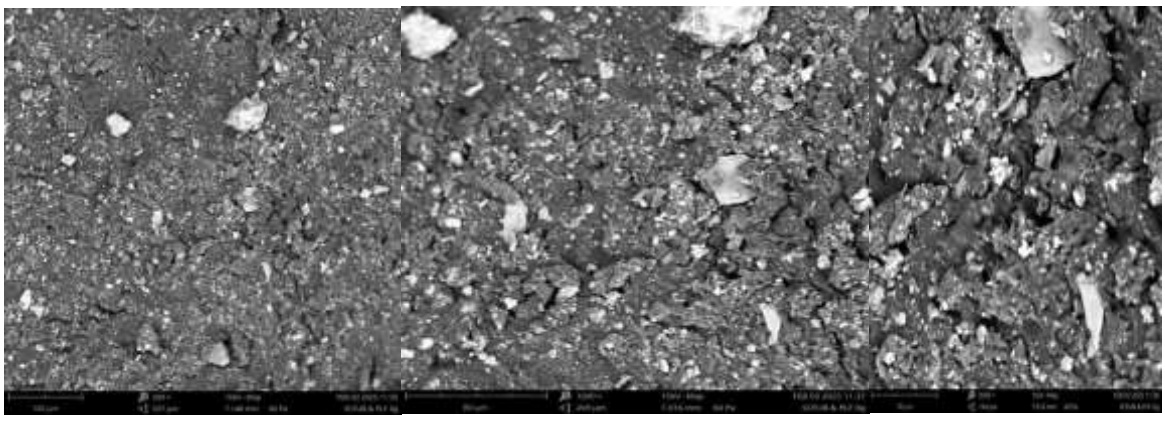

Figure 3: *SEM image for starch composite from 10 g of unripe banana starch containing 0 g of pinapple leaf fibre*

The SEM image reveals a relatively smooth and homogeneous surface morphology, characteristic of a pure starch matrix without reinforcement. The absence of fibre elements results in minimal surface texture and limited structural complexity. The image likely highlights the presence of micro voids and minor cracks, which contribute to the brittle nature of the composite. The smooth morphology correlates with poor mechanical properties. The lack of interlocking fibre structures further explains the composite's inability to effectively distribute stress under load, leading to early failure (Michler, 2008).

3.3 Elemental Analysis

Elemental analysis showing the elemental composition of the natural rubber composites reinforced with 0 g pineapple leaf fibre (PALF). The analysis provides valuable information on the major elements present, such as carbon (C), oxygen (O), and hydrogen (H), which are characteristic of organic polymeric materials.

Table 1: *Shows the elemental composition of starch composite from 10 g of unripe banana containing 0 g of pineapple leaf fibre*

Element Number	Element Name	Atomic Conc.	Weight Conc.
6.0	Carbon	54.28	31.91
14	Silicon	13.73	18.88
26	Iron	05.62	15.37
13	Aluminum	10.58	13.98
20	Calcium	03.21	06.30
7.0	Nitrogen	06.67	04.57
12	Magnesium	02.35	02.79
19	Potassium	01.10	02.11
22	Titanium	0.620	01.45
11	Sodium	0.930	01.05
17	Chlorine	0.460	0.790
16	Sulfur	0.280	0.450
25	Manganese	0.090	0.250
15	Phosphorus	0.080	0.110

Table 1 shows the results of the Energy Dispersive X-ray (EDX) analysis for this sample, revealing a high atomic concentration of carbon (54.28%), which aligns with the organic nature of the starch matrix. The presence of silicon (13.73%), iron (5.62%), and aluminum (10.58%) suggests possible impurities or interactions occurring during the composite fabrication process. Additionally, calcium (3.21%) and magnesium (2.35%) indicate minimal mineral incorporation, likely originating from the extraction process. The relatively limited elemental diversity highlights the absence of reinforcing fibers, which typically contribute additional elemental components to the composite structure.

3.4 Scanning Electron Microscopy (SEM) images of Unripe Banana Starch **(Musa sapientum** L.) Composites Reinforced with 5 g Pineapple Leaf Fibre, (*Ananascomosus L. Merr*.) providing insights into the material's formation process, degree of homogeneity, and the nature of interactions between its constituents.

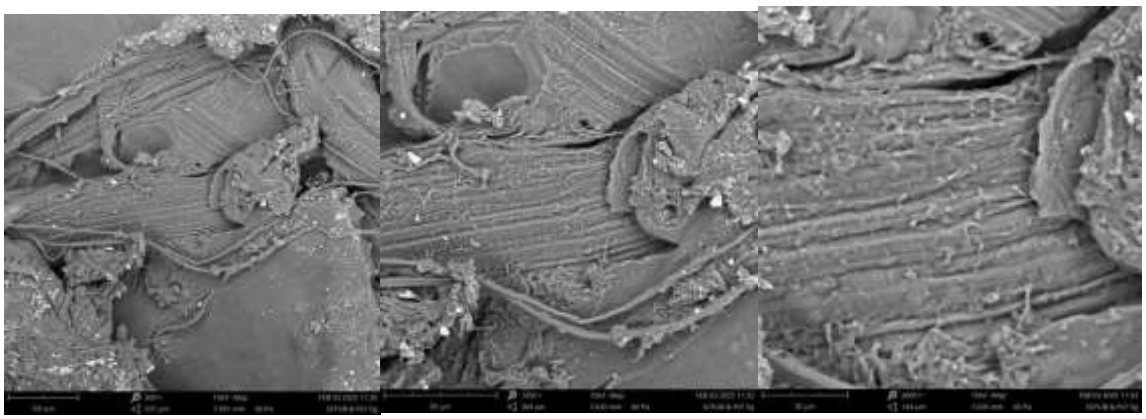

Figure 4: *SEM image for starch composite from 10 g of unripe banana starch containing 5 g of pineapple leaf fibre*

The SEM micrograph of the composite containing 5 g of pineapple leaf fibre (Figure 4) reveals a notably more textured and fibrous surface morphology in comparison to the fibre-free (0 g) sample. Distinctly embedded fibres are observed, forming a dense, interconnected network within the starch matrix. This structural arrangement indicates enhanced interfacial bonding between the fibres and the matrix, which is expected to contribute positively to the composite's mechanical performance. The increased surface roughness observed in the image suggests improved load transfer efficiency and greater resistance to crack initiation and propagation. The fibrous morphology observed is consistent with the superior tensile and compressive properties recorded for the fibre-reinforced composite

3.5 Elemental Analysis

Elemental analysis showing the elemental composition of the natural rubber composites reinforced with 5 g pineapple leaf fibre (PALF). The analysis provides valuable information on the major elements present, such as carbon (C), oxygen (O), and hydrogen (H) which are characteristic of organic polymeric materials.

Table 2: *Shows the elemental composition of starch composite from 10 g of unripe banana containing 5 g of pineapple leaf fibre*

Element Number	Element Name	Atomic Conc.	Weight Conc.
26	Iron	11.15	26.24
6.0	Carbon	40.57	20.53
13	Aluminum	11.23	12.77
20	Calcium	07.54	12.74
7.0	Nitrogen	21.07	12.43
25	Manganese	02.37	05.48
22	Titanium	02.65	05.34
14	Silicon	01.85	02.19
17	Chlorine	01.14	01.71
16	Sulfur	0.420	0.570
15	Phosphorus	0.000	0.000
12	Magnesium	0.000	0.000
11	Sodium	0.000	0.000
19	Potassium	0.000	0.000

Table 2. Shows the result for the EDX analysis for this sample reveals a higher atomic concentration of carbon and additional elemental components introduced by the fibre reinforcement. Carbon remains predominant, reflecting the organic nature of both the starch matrix and the pineapple leaf fibres. Notably, the concentrations of calcium (7.54%) and magnesium (5.48%) increase, suggesting

improved structural stability. Elements such as aluminum (11.23%) and silicon (1.85%) are present in reduced amounts compared to the 0g sample, possibly due to better dispersion and bonding of fibre elements within the matrix. The enhanced elemental diversity correlates with the improved mechanical and thermal properties observed in the composite.

3.6 Thermogravimetric Analysis (TGA)

The TGA were conducted to assess the thermal stability and decomposition characteristics of the natural rubber–pineapple leaf fibre (PALF) composites. The thermograms (Figure 5 and Figure 6) show distinct stages of weight loss associated with different thermal events in the samples.

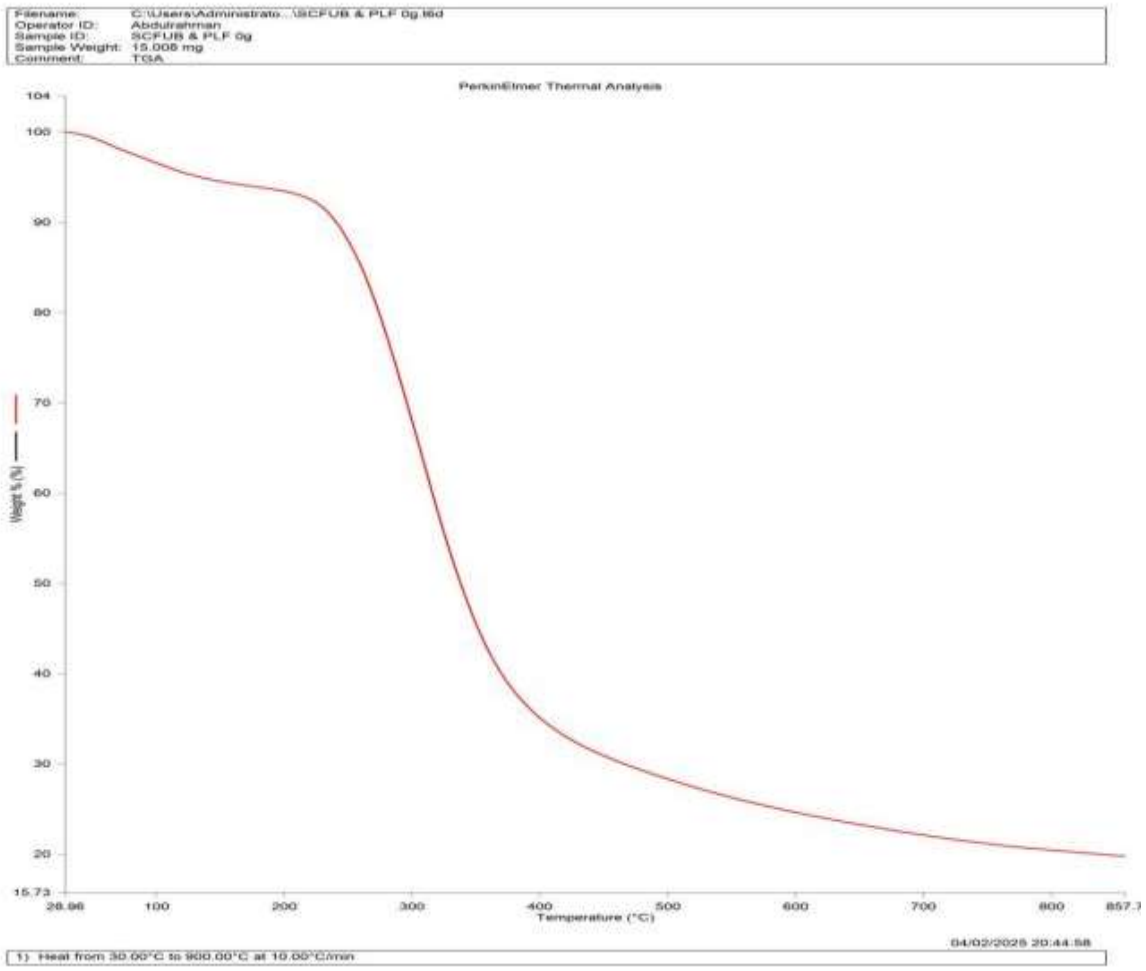

Figure 5: *TGA for starch composite from 10 g of unripe banana starch containing 0 g of pineapple leaf fibre*

Figure 5 shows the TGA curve for the composite without fibre shows a simpler thermal degradation profile compared to the 5 g sample. The initial weight loss between 50°C and 150°C corresponds to moisture evaporation, similar to the fibre-reinforced sample. However, the second weight loss phase, observed between

200°C and 350°C, occurs at a lower temperature range, indicating reduced thermal stability. The absence of fibre reinforcement results in the starch matrix decomposing more rapidly under heat. The lack of a significant residual mass at higher temperatures further underscores the lower thermal resistance of this composite (Nurazzi*et al.*, 2021)

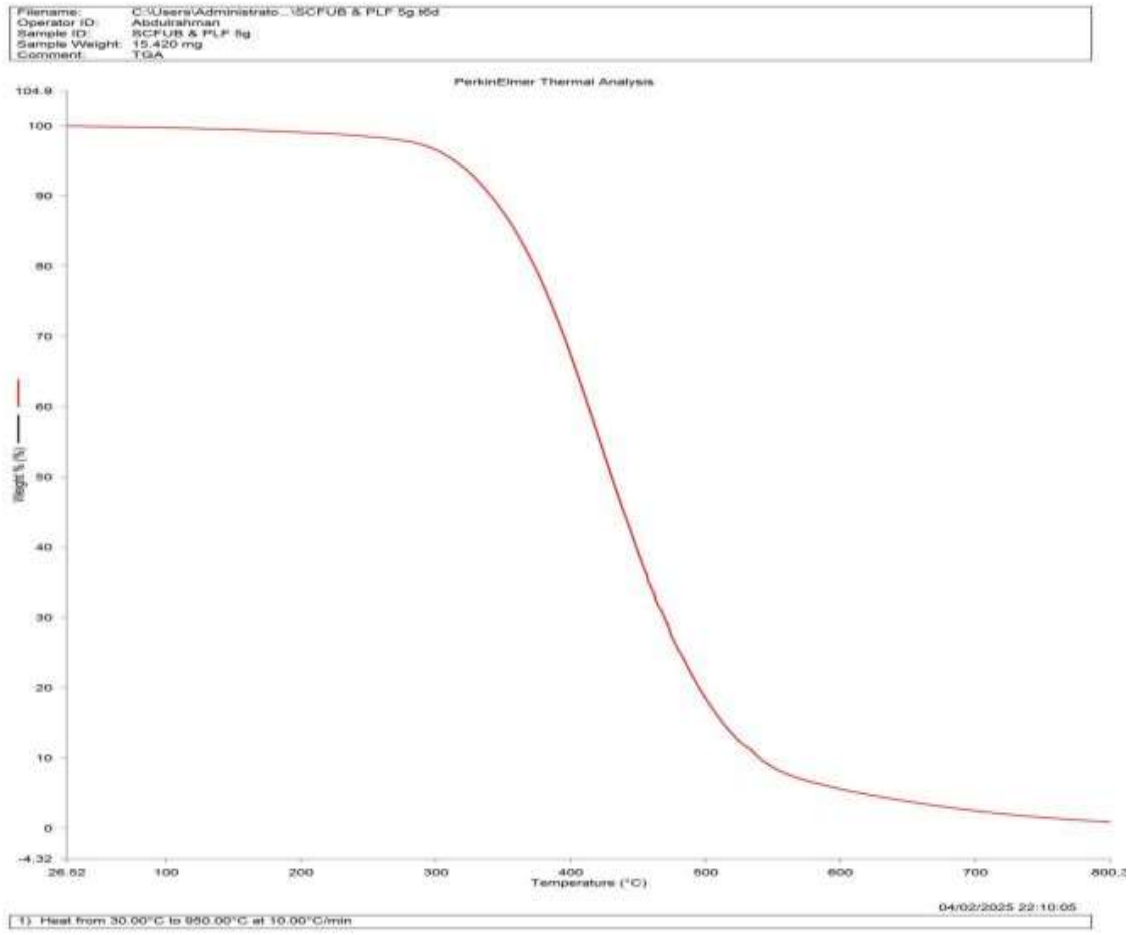

Figure 6: *TGA for starch composite from 10 g of unripe banana starch contaning 5 g of pineapple leaf fibre*

Figure 6 presents the thermogravimetric analysis (TGA) curve for the composite containing 5 g of pineapple leaf fibre, illustrating a characteristic two-step thermal degradation process. The initial mass loss, occurring between approximately 50 °C and 150 °C, is primarily associated with the evaporation of moisture and low-molecular-weight volatile compounds. This stage reflects the composite's inherent moisture content and its thermal behaviour at lower temperatures. The second and more substantial weight loss, observed between 250 °C and 400 °C, corresponds to the thermal decomposition of organic constituents, notably starch and cellulose derived from the incorporated fibres. The inclusion of pineapple leaf fibres appears to enhance the thermal stability of the composite relative to the 0 g fibre sample, likely due to the reinforcing effect of cellulose within the matrix. Furthermore, the greater residual mass at elevated temperatures indicates the

formation of a stable carbonaceous char, which is suggestive of improved thermal resistance and structural integrity at high temperatures (Siregar *et al*., 2011).

3.7 X-Ray Diffraction (XRD) Analysis

X-Ray Diffraction (XRD) analysis were carried out to examine the crystalline structure and phase composition of the samples. This technique provides valuable information on the degree of crystallinity, the presence of amorphous regions and the nature of structural ordering within the material (Figure 7 and Figure 8).

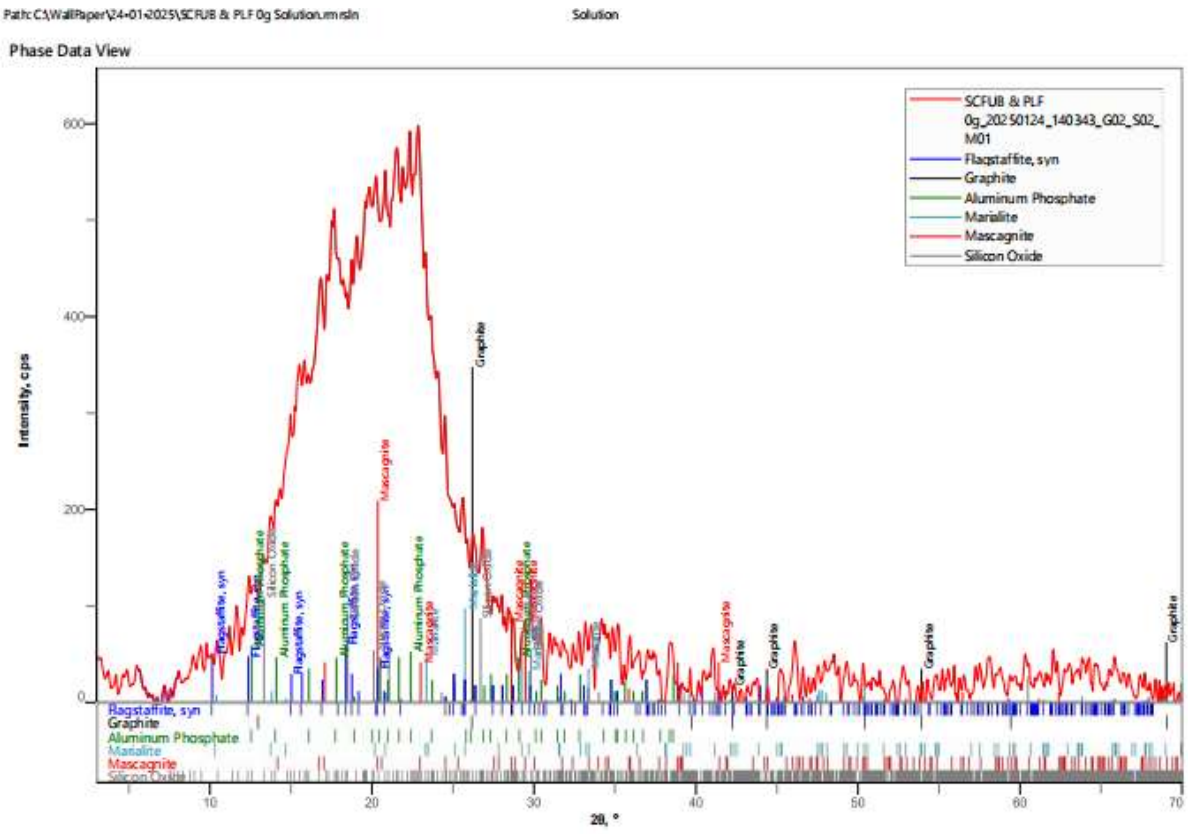

Figure 7: *XRD for starch composite from 10 g of unripe banana starch containing 0 g of pineapple leaf fibre*

Figure 7 shows the XRD pattern for the composite without fiber reveals broad and less intense peaks, characteristic of an amorphous structure. A significant peak is typically observed around 17° to 20° 2θ, corresponding to the semi-crystalline nature of native starch. The broadness of the peaks indicates poor crystalline alignment, which is expected for a pure starch matrix without reinforcing fibers. The absence of sharp and well-defined peaks underscores the limited ordered regions within the composite. This structural characteristic contributes to the brittle nature of the pure starch composite, as observed in mechanical testing results.

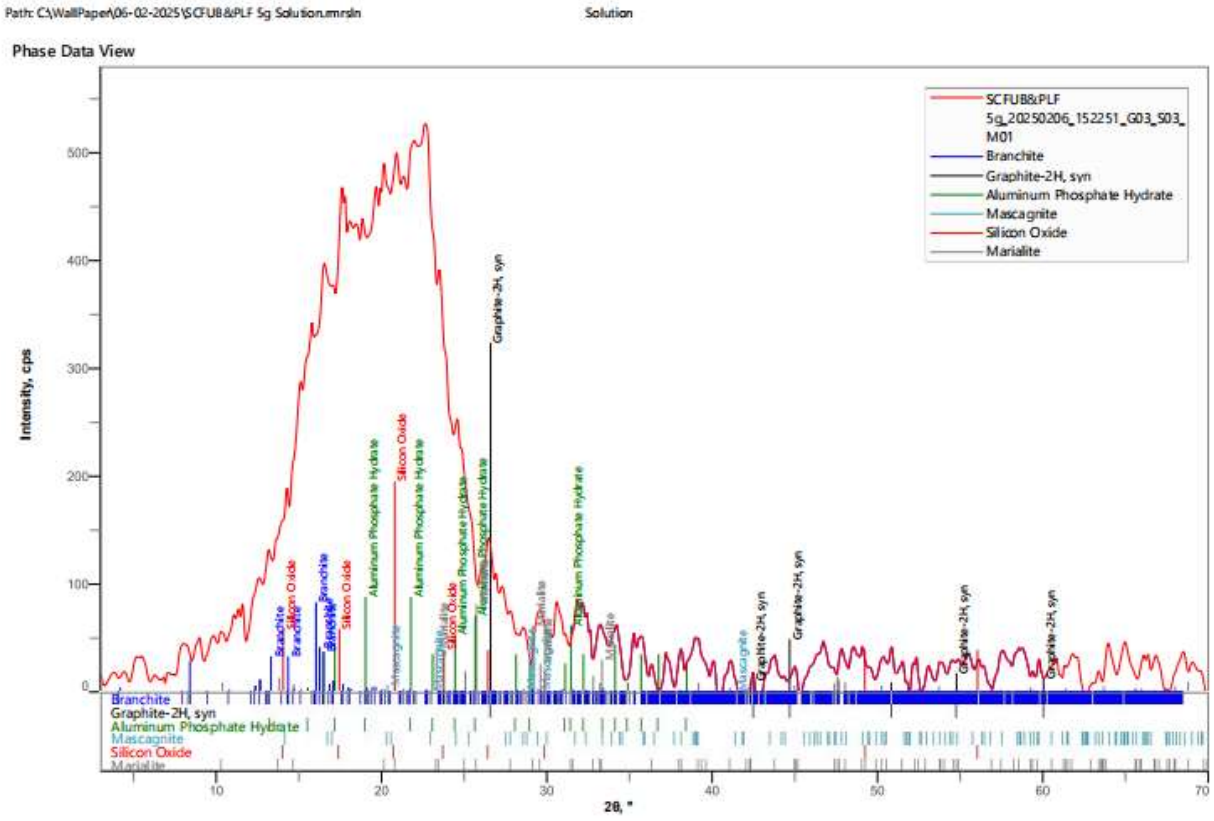

Figure 8: *XRD for starch composite from 10 g of unripe banana starch containing 5 g of pineapple leaf fiber*

The X-ray diffraction (XRD) pattern for the composite with 5 g of pineapple leaf fiber is shown in Figure 8, where significant variations from the fiber-free sample (0 g) are evident. Indicating increased crystallinity in the composite, the diffraction pattern shows sharper and more intense peaks, especially at about 22° and 25° (2θ). The addition of cellulose fibers, which encourage a more ordered molecular arrangement within the starch matrix, is responsible for this increase in crystallinity. The composite's improved mechanical qualities, such as increased tensile strength and thermal stability, are closely linked to its improved crystalline structure. The existence of clear, well-defined peaks additionally implies that fiber reinforcement enhances the structural integrity of the composite by helping to form a more ordered and heat-resistant material.

4. CONCLUSION

This study highlights the potential of biodegradable composites as environmentally friendly substitutes for traditional synthetic materials by successfully developing and characterizing biodegradable composites made from unripe banana starch and pineapple leaf fibers. In fiber-reinforced formulations, structural and morphological analyses verified increased crystallinity, improved mechanical performance, and improved fiber–matrix adhesion. Thermal analysis

also showed that the composites were more stable, and the sample that contained 5 g of pineapple leaf fiber had the best qualities, such as increased mechanical strength, thermal resistance, and structural integrity. These findings demonstrate that fiber-reinforced, starch-based biocomposites are suitable for sustainable uses, especially in the areas of environmentally friendly material innovation and biodegradable packaging.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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