Journal of Pure and Applied Sciences (JPAS),
Volume 2, Issue 2, Page 136-147, 2026

Valorization of Acha Starch-Based Bioplastics Reinforced with Plantain Peel Powder

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ABSTRACT

This study investigates the valorization of Acha starch-based bioplastics reinforced with plantain peel powder. Bioplastic films were prepared via solution casting using glycerol as a plasticizer, with plantain peel incorporated at 10 wt% (SF2) and 20 wt% (SF3), alongside a plasticized starch control (SF1) and a pure starch film (SF4).Fourier Transform Infrared (FTIR) analysis revealed that all samples displayed broad absorption bands in the region of 3200-3400 cm^{-1}, corresponding to O-H stretching vibrations, characteristic of hydroxyl groups present in starch, peaks observed at 2924 cm^{-1} point to possible interactions between starch chains and lignocellulosic constituents of the plantain peel. Scanning Electron Microscopy (SEM) showed increased surface roughness and partial dispersion of filler particles, with slight agglomeration observed at higher filler loading. X-ray diffraction (XRD) patterns exhibited characteristic diffraction peaks at 2θ between 20°-24° with crystallinity indices ranging from 39-55%, indicating that plantain peel altered starch chain organization. TGA showed initial moisture loss below 120°C and major degradation occurring above approximately 250°C, indicating typical starch-based thermal behaviour with filler-dependent variations. The novelty of this work lies in the first reported use of an acha starch-plantain peel composite, highlighting the valorisation of plantain peel waste as a sustainable reinforcement and its structure-property relationships.

Keywords*— Bioplastics, Plantain peel, biodegradable materials, thermal stability, XRD, FTIR, lignocellulosic filler, solution casting, *Digitaria exilis

I. INTRODUCTION

The common use of petroleum-based plastics has resulted in significant environmental challenges, such as persistent pollution, greenhouse gas emissions, and dependence on non-renewable resources. Conventional plastics pose serious ecological and human health risks arising from slow degradation periods and accumulation in landfills, soils, and aquatic ecosystems [1]. Consequently, there has been a consistently growing global interest in the development of renewable biodegradable materials as alternatives to synthetic plastics.

Bioplastics, a form of plastic made from natural polymers have emerged as promising alternatives to conventional plastics [2]. The starch polymer is popular due to its abundance, low cost, biodegradability, non-toxicity, and film-forming capability. Bioplastics are typically produced through gelatinisation and plasticisation processes that disrupt the native crystalline structure of starch granules and allow the formation of a continuous polymeric matrix [3]. A suitable amount of a plasticizer such as glycerol is added to the matrix because of its ability to form hydrogen bonds with starch chains, increasing chain mobility and flexibility while reducing brittleness.

Plant based fillers have been used in recent years to improve the properties of starch-based bioplastics. These fillers enhance mechanical properties, thermal stability and barrier performance while reducing the overall material cost of producing bioplastics [4].

Acha (*Digitaria exilis*.) is an annual tropical grass cultivated primarily in West Africa for its starch-rich, tiny seeds [5]. The cereal is valued for its high digestibility, gluten-free nature, and rapid cooking time. It is drought-resistant and well adapted to poor soils, making it an important food security crop in semi-arid regions [6]. However, compared with major starch sources like corn starch, cassava starch, and potato starch, acha remains underutilized and under-researched in starch-based bioplastics. Most studies focus on its food and nutritional applications, with limited work on polymer or biocomposite development.

Agricultural waste materials offer additional opportunities for sustainable materials development. Plantain peel is rich in cellulose, hemicellulose, lignin and inorganic minerals, thereby making it suitable for use as a reinforcing filler in polymer composites [7], [8]. Previous studies have demonstrated that lignocellulosic fillers can improve stiffness, tensile strength and thermal stability of starch-based films by enhancing interfacial interactions and restricting polymer chain mobility [9].

This research work which involves exploring acha starch as a bioplastic feedstock presents an opportunity to expand the raw material base for sustainable polymers. Additionally, limited work has examined the combined effects of glycerol and plantain peel in underutilised starch systems such as acha. This research works fills the gap in knowledge by simultaneously investigating the functional, structural, morphological and thermal characteristics of Acha starch bioplastics reinforced with agricultural waste fillers. The understanding gained is essential for optimising material performance and identifying suitable application areas.

Therefore, the present study focuses on the valorization of bioplastics derived from Acha (*Digitaria exilis*.) starch using glycerol as a natural plasticizer and plantain peel powder as a sustainable filler.

Comprehensive characterisation was conducted using Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and thermogravimetric analysis (TGA) to elucidate chemical interactions, structural organisation, surface morphology and thermal stability. This work contributes to research in green chemistry and sustainable materials research by demonstrating the potential of acha starch and plantain peel as renewable resources for the development of biodegradable bioplastics.

II. MATERIALS AND METHODS

A. Materials

Acha (*Digitaria exilis*) grains were obtained from a local market in Nigeria. Glycerol (Loba Chemie PVT) and glacial acetic acid (BDH laboratory supplies) were purchased from a standard chemical supplier in Ibadan and used without further purification. Fresh plantains (Musa paradisiaca) were obtained from Ago-Aduloju market and the peels were thoroughly washed with distilled water and air-dried. All materials employed in this study were selected based on their availability, low toxicity and environmental compatibility.

B. Extraction of amorphous silica from groundnut shell ash (GSA)

Fresh plantain peels were washed repeatedly with distilled water to remove surface impurities and adhering dirt. The peels were then cut into small pieces and oven-dried at 60 °C for 24 h until a constant weight was obtained. The dried peels were ground using a laboratory grinder and sieved to obtain a fine powder with particle size less than 250 µm. The resulting plantain peel powder was stored in airtight containers prior to use.

C. Extraction of Acha Starch

Acha grains were finely ground using an electric mill. 500 grams of the milled flour was mixed with 1L of NaOH (0.1% w/v) in order to enhance removal of proteins and non-starch impurities. The mild alkaline condition was chosen to reduce possible structural alteration of starch granules while improving starch purity for bioplastic formulation. The mixture was kept at 40C for 18h, after it was homogenized in an industrial blender and passed first through cheesecloth to remove fibers and then through a 100-mesh sieve (150µm). The suspension was allowed to sit for 18h. The supernatant was discarded and the retained solids was washed three times to remove NaOH. The obtained starch was then placed into plastic cylindrical trays (16 × 13 × 6 cm) and dried at 40 0C for 24h. Finally, the starch was ground in a mortar, passed through a 100-mesh sieve and stored in a sealed plastic bag at 25 0C until use [4]. The starch yield

was calculated as the percentage ratio of the dried starch obtained to the initial weight of acha grains used. The extraction yielded 36.2% starch under applied laboratory conditions.

D. Preparation of Acha Starch-Based Bioplastics

Bioplastic films were prepared using a solution-casting technique. Acha starch (10g) was dispersed in 50 mls of distilled water under continuous stirring. Glycerol was added as a plasticizer at a fixed concentration (20%), while plantain peel powder was added as a natural filler. The mixture was heated to 80-90 °C with constant stirring to induce starch gelatinisation and ensure homogeneous dispersion of all components. 5ml of acetic acid solution (5%) was added to speed up gelatinisation and improve film homogeneity.

The resulting viscous paste was cast onto levelled glass plates and dried at room temperature for 48 h. The dried films were carefully peeled off, followed by heating again in an oven at 85 0C for 2 hrs. The bioplastic was then allowed to cool down properly before storing them for characterisation. The thickness of the bioplastic was measured using a micrometer at multiple points across the sample before analysis. The average thickness values recorded for the samples were SF1 = 0.12mm, SF2 = 0.14mm, SF3 = 0.16mm and SF4 = 0.13mm. Table 1 shows the composition of the bioplastic.

E. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR analysis was performed using an Agilent Cary 630 FTIR in the wavenumber range of 4000-400 cm^{-1}. Samples were analysed using the attenuated total reflectance (ATR) method. The spectra were recorded to identify functional groups and assess intermolecular interactions between starch, glycerol and plantain peel filler.

Table 1: Bioplastic Composition

Sample ID	Starch (g)	Plantain peel (%)	Glycerol (%)	Acetic acid (ml)
SF1	10	0	20	5
SF2	9	10	20	5
SF3	8	20	20	5
SF4	10	0	0	5

F. X-ray Diffraction (XRD) Analysis

XRD measurements were carried out using a Rigaku MiniFlex X-ray diffractometer equipped with Cu Kα radiation (λ = 1.5406 Å). Diffraction patterns were recorded over a 2θ range of 5-40° at room temperature. The crystallinity and structural organisation of the bioplastic films were analysed by evaluating peak positions, intensities and amorphous background contributions. The crystallinity index was calculated using the Segal method, where the index of crystallinity Ci (%) is obtained by the following equation

$$\mathrm{CI}(\%) = \left(\frac{\mathrm{I002}-\mathrm{Iam}}{\mathrm{I002}}\right) * 100 \qquad (1)$$

Where:

I002 = Intensity of the crystalline peak, and

Iam = Intensity of the amorphous peak

G. Scanning Electron Microscopy (SEM)

The surface morphology of the bioplastic films was examined using a Phenom ProX desktop scanning electron microscope (SEM) with an accelerating voltage of 15kV. Samples were mounted on aluminium stubs and sputter-coated with a thin layer of gold to improve conductivity. A SEM images were obtained at appropriate magnifications to assess surface uniformity, filler dispersion and microstructural features.

H. Thermogravimetric Analysis (TGA)

Thermal stability of the bioplastic films was evaluated using thermogravimetric analysis on a PerkinElmer TGA 4000 Thermo Gravimetric Analyzer. Approximately 5-10 mg of each sample was heated from room temperature to 600°C at a heating rate of 10 °C min^{-1} under a nitrogen atmosphere. Weight loss profiles were recorded to assess thermal degradation behaviour.

In all formulations, the total dry mass was kept constant at 10g. Plantain peel filler was introduced as a partial substitution of starch, rather than as an additional independent component, in order to maintain a constant solids basis across all samples.

III. RESULTS AND DISCUSSION

A. Fourier Transform Infra-Red Spectroscopy (FTIR)

FTIR spectra of acha starch bioplastic are shown in Figure 1. All samples displayed broad absorption bands in the region of 3200-3400 cm^{-1}, corresponding to O-H stretching vibrations, characteristic of hydroxyl groups present in starch.

A broadening and slight shifting of this band in plasticised and reinforced samples were observed, indicating the formation of extensive hydrogen bonding interactions among the components. The peak observed around 2920 cm^{-1} corresponds to C-H stretching vibrations, while peaks in the region of 1640-1650 cm^{-1} correspond to absorbed water and hydrogen-bonded structures. The presence of peaks between 1000 and 1150 cm^{-1}, associated with C-O-C and C-O stretching vibrations, confirms the polysaccharide backbone of the starch matrix. Variations in absorption peak intensity were observed in samples containing plantain peel at 2924 cm^{-1}, which suggest possible interactions between starch chains and lignocellulosic constituents of the plantain peel [10], [11].

Overall, the FTIR results confirm that glycerol acts as an effective plasticizer by promoting hydrogen bonding, while plantain peel contributes to intermolecular interactions that influence the structural integrity of the films.

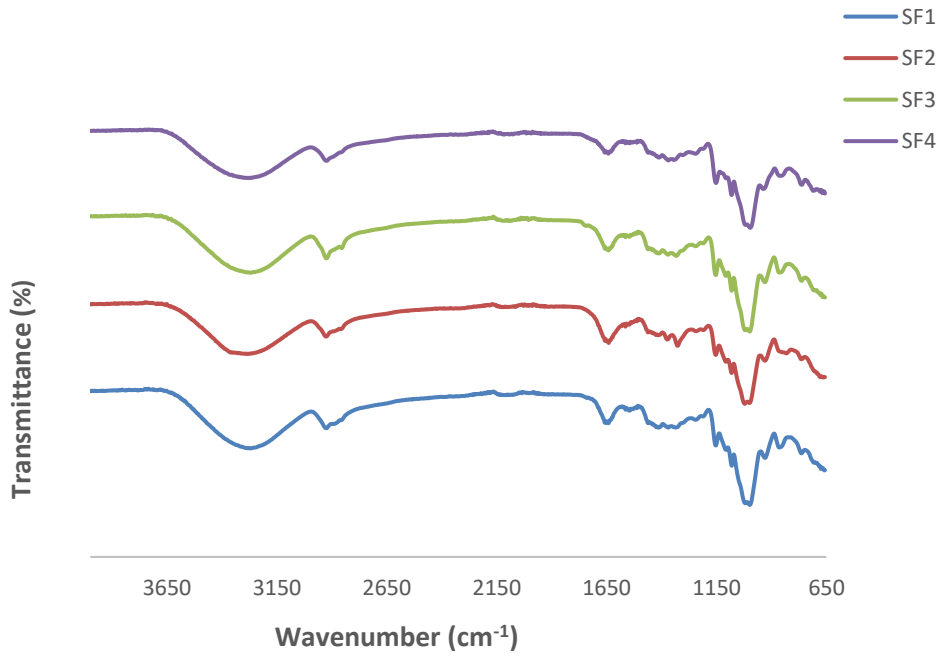

Figure 1: FTIR spectra of acha starch- based bioplastic films with varying plantain peel content

B. X-Ray Diffraction Analysis

X-ray diffraction analysis was conducted to investigate the structural organisation of the acha starch-based bioplastics. The XRD spectra are shown in figure 2. All the samples had a predominantly amorphous diffraction pattern indicated by a broad halo in the 2θ range. Pure starch bioplastic (SF4) had a unique diffraction pattern with peak at 21.4°, with a crystallinity index (CI) value of 42.67%. Conversely, the plasticized starch bioplastic SF1 had a diffraction pattern with peak at 24.3° and the lowest CI value of (39.29%). This is likely due to the glycerol replacing the inter- and intra-molecular hydrogen bonds which has disrupted the crystallinity of starch during the bioplastic film fabrication [12], [13].

However, addition of filler changed the diffraction pattern with samples SF2 and SF3 exhibiting a weak and broader diffraction pattern with peaks at 20.87° and 20.74° respectively. These samples showed higher CI values of 55.26% and 50.00%, respectively. The higher CI values observed for SF2 and SF3 suggest that the incorporation of plantain peel filler promoted a greater degree of structural ordering within the starch matrix. This finding is consistent with reports that cellulose-containing lignocellulosic fillers can influence the crystallinity of starch-based biocomposites [14], [15].

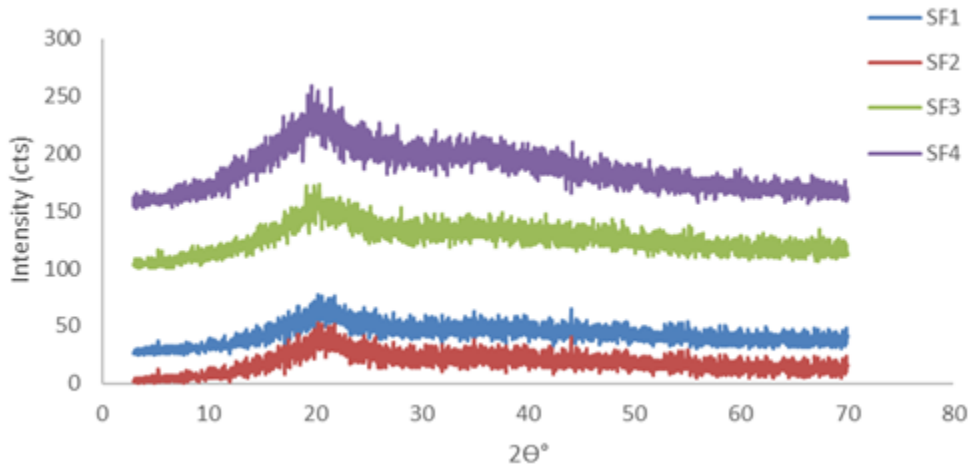

Figure 2: XRD spectra showing the crystalline structure of Acha starch bioplastic films at different plantain peel loadings (a) SF1 (b) SF2 (c) SF3 and (d) SF4.

Table 2: Crystallinity Index (CI) values

Sample	2θ Peak (°)	I_{200} (cts)	I_{am} (cts)	CI (%)
SF1	24.3	28	17	39.29
SF2	20.87	38	17	55.26
SF3	20.74	60	30	50.00
SF4	21.4	75	43	42.67

C. Scanning Electron Microscopy (SEM) Analysis: Morphology and Filler Dispersion

The surface morphology of the bioplastic films was examined using scanning electron microscopy with a magnification of 200x is shown in figure 3a-d. In figure 3(a), the SEM micrographs of glycerol-plasticised Acha starch films show a relatively smooth and continuous surfaces. This is an indication of good film-forming ability and effective starch gelatinisation. However, minor surface irregularities were observed, which could be attributed to moisture loss during drying.

The incorporation of plantain peel powder in figure 3(b) and (c) resulted in noticeable changes in surface morphology. At 10% filler content, the peel particles appeared to be well dispersed within the starch matrix, with minimal agglomeration, suggesting good interfacial interaction between the filler and polymer. At 20% filler levels, slight surface roughness and particle protrusions were observed, which may influence water interaction behaviour. Bioplastics made with fillers had fewer holes and cracks which shows a relative compatibility between filler and the starch granules. This is similar to reports by [11]. In both cases, the surface is smoother than the bioplastic whose micrograph is shown in figure 3(d) which is a bioplastic in which both filler and plasticizer are absent. The bioplastic becomes fragile with a rough surface as described in research by [16]. The observed cracks could be attributed to the starch bonds α-1,4-glucosidic connection, which was more amorphous [17]. Overall, SEM analysis confirms that plantain peel can be effectively incorporated into acha starch bioplastics, contributing to structural modification without severe phase separation.

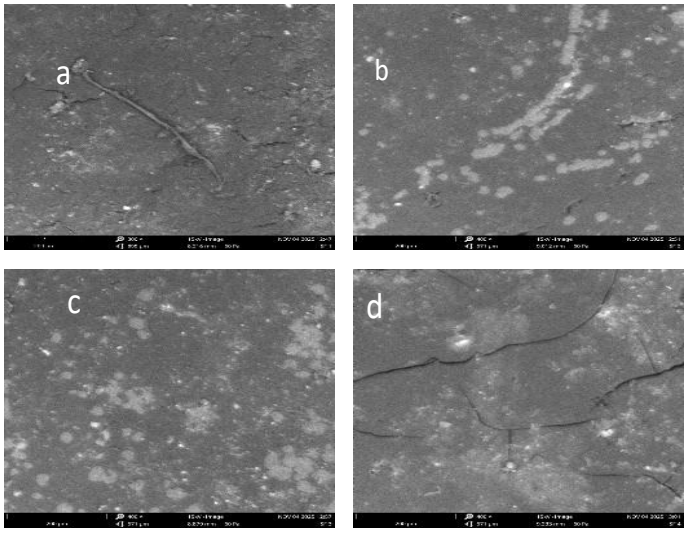

Figure 3: SEM micrographs showing surface morphology of Acha starch bioplastic films at different plantain peel loadings (a) SF1 (b) SF2 (c) SF3 and (d) SF4

D. Thermal Properties

Thermogravimetric analysis (TGA) of starch bioplastic and composite bioplastic decomposition graphs is shown in Fig 4a-b. The thermal stability of starch-based bioplastic formulations was investigated using TGA and DTG. Four formulations were evaluated: SF1 (glycerol + starch), SF2 (10 wt% plantain peel), SF3 (20 wt% plantain peel), and SF4 (neat starch). SF4 retained 98.55 wt% of its original mass at 100 °C, corresponding to a mass loss of about 1.45 wt% mainly due to moisture and volatile removal. It also showed a slower degradation rate at higher temperatures (DTG = - 0.093 %/min at 220 °C) with a decomposition onset around 225 °C, which indicates a relatively higher thermal stability. This is consistent with Tan et al. (2022), who reported starch degradation between 220 and 385 °C [13]. In comparison, SF1 retained 98.67 wt% at 100 °C (about 1.33 wt% mass loss), indicating slightly lower initial mass loss. SF1 also showed an additional mass-loss region between 100-150 °C, likely due to glycerol and bound water evaporation, and a lower decomposition onset at about 210 °C, suggesting reduced thermal stability compared to SF4.

The TGA results of the plantain peel composites show a mixed thermal behaviour depending on temperature. SF2 (10 wt% peel) exhibited relatively stable behaviour below 200 °C, with reduced early-stage mass loss, suggesting improved resistance to moisture and plasticizer evaporation. However, above 200 °C it degraded rapidly (DTG = -0.590 %/min at 220 °C), indicating lower thermal stability at higher temperatures. In contrast, SF3 (20 wt% peel) showed lower degradation rates at lower temperatures (DTG = -0.036 %/min at 200 °C), which could be attributed to filler dilution effects, but also followed the general trend of reduced stability at elevated temperatures. These results agree with Castañeda-Niño et al. (2025) [18], who reported that plantain peel fibres can reduce thermal stability due to hemicellulose content.

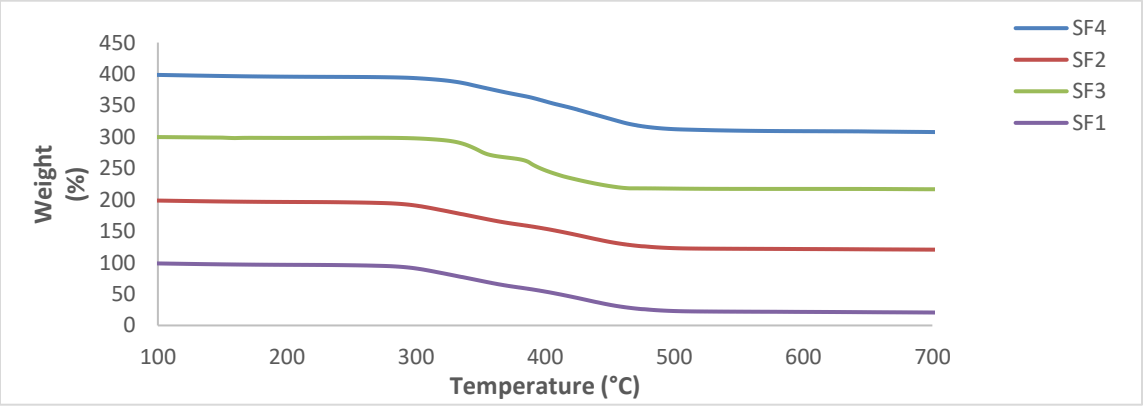

Figure 4a: Thermogravimetric Analysis (TGA) curves of Acha starch-based bioplastics (SF1-SF4)

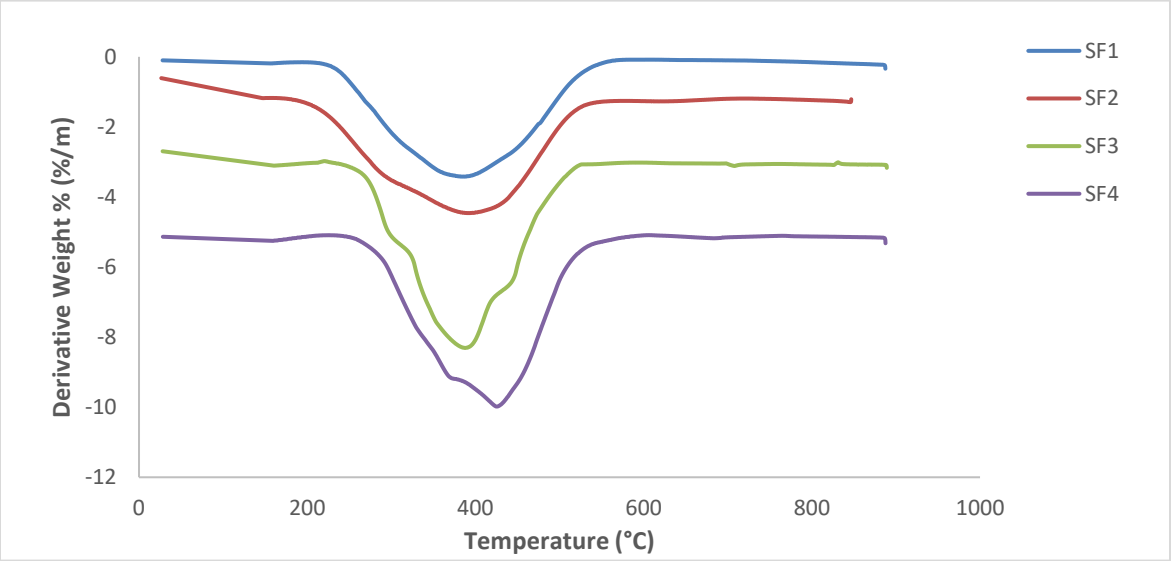

Figure 4b: Derivative Thermogravimetric (DTGA) curves of Acha starch-based bioplastics (SF1-SF4)

IV. CONCLUSION

This study developed acha starch-based bioplastics reinforced with plantain peel powder and demonstrated how filler addition influences their structural, morphological, and thermal properties. XRD results showed characteristic peaks at 2θ ≈ 17°, 20°, and 22°, with crystallinity indices ranging from 39-55%, indicating changes in starch chain arrangement due to plantain peel incorporation. FTIR analysis revealed a shift in the O-H stretching band from about 3280 to 3250 cm^{-1}, confirming interactions between starch, glycerol, and plantain peel components. SEM images showed better filler dispersion and matrix compatibility at moderate loading, while higher filler content led to increased surface roughness and some agglomeration.

TGA showed a small initial mass loss of about 1-1.5% below 120 °C, followed by major degradation between 250 and 300 °C. Plantain peel improved resistance to low-temperature mass loss, but samples containing higher filler levels degraded more rapidly above 200 °C, showing that its effect on thermal stability is dependent on concentration rather than a uniform improvement.

This work also demonstrates the potential of using plantain peel waste as a sustainable reinforcement for acha starch bioplastics. It is also the first known report of an acha starch-plantain peel composite system, contributing to agricultural waste valorisation and showing how filler content affects bioplastic structure

and performance. Importantly, this work highlights the effective valorization of plantain peel waste, transforming an abundant agricultural by-product into a value-added functional material for bioplastic reinforcement. This valorization approach not only improves material performance but also contributes to waste reduction and sustainable resource utilization.

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