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Production of bioplastic from waste agricultural biomass

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ARTICLE INFO	ABSTRACT
	This study investigates the production of bioplastics from plant waste materials, specifically bean peel, plantain peel, and wooden wine cork, using an acid-alkaline pulping method to extract cellulose. The extracted cellulose was bleached, and its purity was confirmed through Fourier-transform infrared (FTIR) spectroscopy. Bean peel showed the highest cellulose yield (45.5%), followed by plantain peel (38.0%) and wooden wine cork (20.0%). The cellulose was converted into cellulose acetate via acetylation, with bean peel yielding 80% cellulose acetate. FTIR analysis revealed significant functional groups, and the degree of substitution for cellulose acetate ranged from 1.25 to 2.76. Biodegradation tests demonstrated significant weight reduction in the bioplastics over four weeks, confirming their biodegradability. This research underscores the potential of using plant waste as a sustainable raw material for bioplastic production, contributing to waste management and the reduction of plastic pollution. **Keywords:** bioplastics, agricultural biomass, cellulose extraction, sustainability, biodegradation, plastic pollution

INTRODUCTION

Plastic pollution has emerged as a significant environmental issue, contributing to ecological degradation and posing threats to human health and wildlife (Rochman et al., 2013). The widespread use of conventional plastics, which are primarily derived from fossil fuels, has led to increased greenhouse gas emissions and resource depletion (Awasthi et al., 2022). In response to these challenges, there is a growing interest in bioplastics, which are produced from renewable biomass sources and offer a more sustainable alternative (Thompson et al., 2004).

Waste agricultural biomass, such as fruit peels, plant residues, and wooden materials, represents a substantial and underutilized resource that can be harnessed for bioplastic production. Utilizing these materials not only addresses the issue of agricultural waste disposal but also contributes to the circular economy by converting waste into valuable products (Siracusa et al., 2008). Various methodologies have been explored for the extraction of cellulose from agricultural waste, including acid-alkaline pulping, which enhances the purity and yield of cellulose (Nasir et al., 2017).

Cellulose can then be transformed into bioplastics through acetylation, a process that modifies the polymer structure to improve its properties (Mohanty et al., 2002). This transformation has the potential to create biodegradable plastics that mitigate plastic pollution and support sustainable development goals (Arrieta et al., 2017). The current study investigates the production of bioplastics from waste agricultural biomass, focusing on the extraction of cellulose and its conversion into biodegradable materials. This research aims to contribute to sustainable waste management practices and the development of eco-friendly alternatives to conventional plastics.

MATERIALS AND METHODS

Biomass Collection, Preparation and Drying

Agricultural waste materials, including white bean peels, plantain peels, and wooden wine corks (**Figure 1**), were collected from various local sources in Irete, Owerri West, Imo State, Nigeria. The bean and plantain peels were sourced from Chopii Restaurant, Irete, while the wooden wine corks were obtained from Metro's Event Centre along Onitsha-Owerri Road. Each sample was placed in well-labeled polyethylene bags to prevent contamination and ensure proper handling during transport to the research laboratory.

Upon arrival at the laboratory, the samples were thoroughly washed with distilled water to remove dirt and impurities. The cleaned materials were sun-dried for seven days to achieve substantial moisture reduction. Following this, the samples were subjected to oven drying at 60°C for 24 hours to ensure complete removal of residual moisture. This

Figure 1. Pictures of plant waste materials undergoing drying (a- beans peel, b- plantain peel, c- wooden wine cork) (Source: Field study)

Figure 2. Pictures of plant waste materials after grinding (a- ground beans peel, b- ground plantain peel, c- ground wooden wine cork) (Source: Field study)

combination of sun drying and oven drying was crucial for preparing the biomass for subsequent chemical treatments.

Grinding

The dried materials were ground into fine powder (**Figure 2**) using a mechanical grinder to increase the surface area, facilitating the efficiency of subsequent chemical treatments during cellulose extraction. The ground samples were then sieved using a 100-mesh sieve to ensure uniform particle size, which is critical for consistent chemical interactions during the extraction process.

Cellulose Extraction

The acid-alkaline pulping method, with modifications, was used for cellulose extraction from biomass (Nasir et al., 2017). A 30 g sample of each biomass was placed into a Soxhlet extractor and dewaxed using a benzene/ethanol mixture (2:1 v/v) at 70 °C for 5 hours. The dewaxed biomass was then washed with ethanol for 30 minutes to remove residual oil and wax.

After dewaxing, 5 grams of defatted biomass were treated with 200 ml of 1M HCl at 70°C for 2 hours with continuous stirring to remove hemicellulose. The mixture was filtered using Whatman No. 1 filter paper, and the residue was washed with distilled water until a neutral pH was achieved. The residue was then air-dried overnight. The lignin-rich residue was subsequently treated with 100 ml of 17.5% NaOH (w/v) at 70°C for 2 hours to extract cellulose. The mixture was filtered again using Whatman No. 1 filter paper, and the residue was

washed with distilled water until a neutral pH was achieved, followed by air drying.

The method of Bolat et al. (2023) was used in bleaching, 2.5 grams of the extracted cellulose were treated with 30 ml of 1% NaOH and 20% H_2O_2 (1:2 v/v) at 50°C for 45 minutes. The bleached cellulose was washed with distilled water and oven-dried at 60°C until a constant weight was obtained. The cellulose yield was calculated as the percentage of cellulose obtained from the original biomass sample using the formula by Klunklin et al. (2023):

$$\text{Cellulose yield (\%)} = \frac{\text{weight of cellulose}}{\text{weight of the original sample}} \times 100 \quad (1)$$

Fourier- Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy was used to characterize the functional groups present in the extracted cellulose from each biomass source. A Thermo Nicolet iS50 FTIR spectrometer was used for the analysis, scanning the cellulose samples over a wavelength range of 4000–400 cm^{-1}. The resulting spectra were analyzed to confirm the presence and purity of the cellulose and identify key functional groups, such as aromatic rings, hydroxyl groups, and ethers.

Cellulose Acetate Synthesis

The synthesis of cellulose acetate (CA) was performed following the method described by Battisti et al. (2019) with slight modifications. A sample of 5 g from each raw material

was utilized. To initiate the reaction, 25 ml of acetic anhydride, 25 ml of glacial acetic acid, and 0.1 ml of sulfuric acid were mixed thoroughly. This mixture was gradually added to the cellulose sample, which was then stirred and placed in a water bath at 37°C for 1 hour.

After this initial incubation, an additional 25 ml of acetic anhydride was introduced while stirring continuously. The reaction temperature was then raised to 45°C, and the mixture was stirred for 45 minutes. Subsequently, the temperature increased to 60°C, and a mixture of 50 ml of 70% acetic acid and 0.1 ml of sulfuric acid was added gradually. The solution was left to stir for 30 minutes to maximize the acetylation process.

Upon completion of the acetylation, the solution was removed from the water bath, and 25 ml of distilled water was added, resulting in the formation of a whitish precipitate. An additional 200 ml of distilled water was then added to the mixture, which was stirred and allowed to sit for several minutes. The resulting solution was filtered, and the residue was washed with distilled water until neutral. The obtained CA was dried in an oven at 60°C for 24 hours and then weighed. The yield of CA was calculated using a modified version of the formula described by Klunklin et al. (2023):

$$Yield\ of\ CA\ (\%) = \frac{weight\ of\ CA}{weight\ of\ cellulose} \times 100 \qquad (2)$$

Characterization of Cellulose Acetate

The characterization of cellulose acetate (CA) involved analyzing its functional groups, determining the degree of substitution (DS), and evaluating its solubility, following standard methods.

Degree of substitution

The degree of substitution (DS) refers to the average number of hydroxyl groups on a polymer chain that have been replaced by substituent groups during a chemical modification process. For cellulose acetate, the DS indicates the number of hydroxyl groups on the cellulose molecule that have been substituted by acetate groups, ranging from 0 (no substitution) to 3 (full substitution, where all hydroxyl groups are replaced) (Battisti et al., 2019).

The DS of cellulose acetate was determined using the potentiometric back-titration method described by Candido et al. (2017). For this procedure, 0.1 g of dry cellulose acetate was accurately weighed and transferred into a 250 mL Erlenmeyer flask. Subsequently, 5 mL of 0.25 M sodium hydroxide (NaOH) and 5 mL of 75% (v/v) aqueous ethanol were added to the flask. The mixture was sealed and allowed to stand at room temperature for 24 hours.

After this period, the titration was conducted with a standard 0.25 M NaOH solution, using phenolphthalein as an indicator. This procedure was performed in triplicate to ensure accuracy.

The degree of acetylation- a measure of the percentage of hydroxyl groups on a polymer chain replaced by acetyl groups (Martins et al., 2024)- and the DS were calculated using the following equations provided by Candido et al. (2017):

$$\%\ Acetyl = \frac{\{[(Vbi + Vbt) * \mu b - (Va * \mu a)] * M * 100\}}{mac} \qquad (3)$$

And:

$$DS = \frac{(3.82\ x\ \%Acetyl)}{(102.4 - \%\ Acetyl)} \qquad (4)$$

Where:

Vbi = Volume of NaOH added to the system (L)

Vbt = Volume of NaOH spent in titration (L)

μb = NaOH concentration (M)

Va = Volume of HCl added to the system (L)

μa = HCl concentration (M)

M = Molar weight of acetyl group (43 g mol−1)

Mac = weight of cellulose acetate sample (g).

Solubility test

The solubility test is considered one of the methods for identifying biopolymers. The solubility of cellulose acetate can provide insight into its classification (Jassem et al., 2020). During the experiment, 0.3g of cellulose acetate was immersed in various solvents, namely acetone, dichloromethane, and a mixture of dichloromethane and acetone in a 1:1 volume-to-volume ratio. The immersion process took place at room temperature with continuous stirring for 5 minutes. The percentage of solubility was calculated using the following equation (Jassem et al., 2020).

$$Solubility\ \% = \frac{M1 - M2}{M1} \qquad (5)$$

Where:

M_1 = total mass of cellulose acetate

M_2 = mass of insoluble cellulose acetate

Bioplastic Synthesis

The synthesized cellulose acetate was further processed into bioplastic films. Cellulose acetate was dissolved in a mixture of dichloromethane and ethanol (1:1 v/v) to form a homogeneous solution. The cellulose film was subsequently plasticized by adding glycerol and palm kernel oil, which improved its flexibility and mechanical properties. The bioplastic films were air-dried at room temperature for 48 hours. The solution was poured into Petri dishes and left to dry at room temperature, forming thin bioplastic films. These films were carefully peeled off and stored for further testing, including biodegradability and mechanical properties.

Characterization of Bioplastic

The films' biodegradability was evaluated by burying samples in soil and measuring the mass loss over a 4-week period.
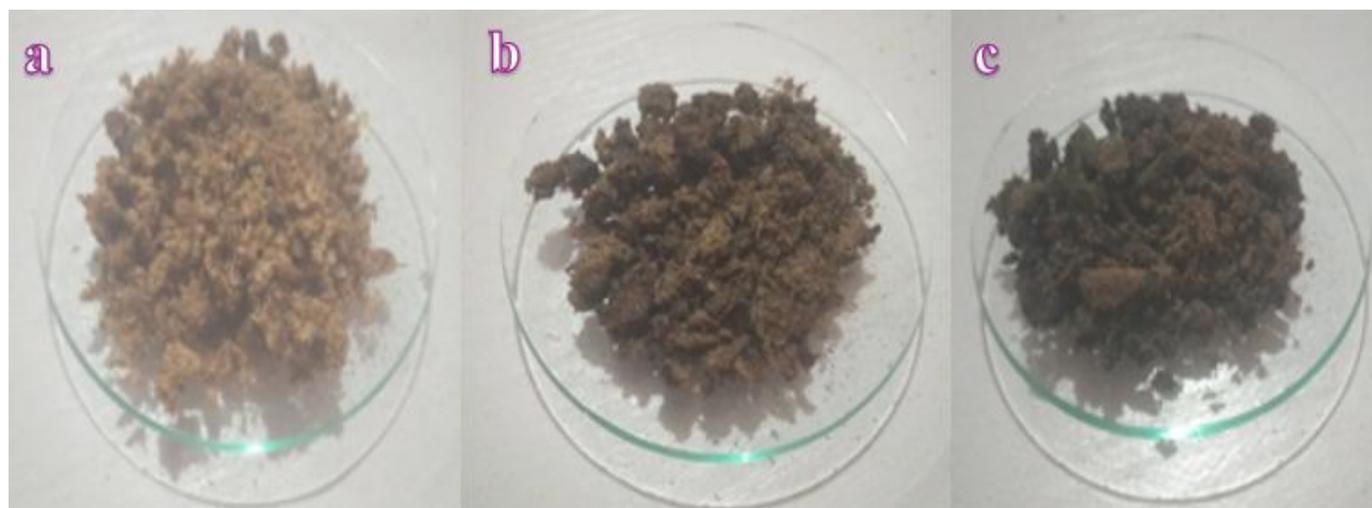
Figure 3. Pretreated plant waste materials (a- pretreated beans peel, b- pretreated plantain peel, c- pretreated wooden wine cork) (Source: Field study)

Figure 4. Bleached cellulose samples (a- beans peel, b- plantain peel, c- wooden wine cork) (Source: Field study)

RESULTS AND DISCUSSION

Extraction of Cellulose

The dewaxing of the various plant materials using acid-alkaline pulping pretreatment method resulted in the removal of waxy substances and lipids from the plant waste materials (**Figure 3**). The pulp obtained exhibited a dark colour because of cell wall breakdown and insufficient elimination of lignin (Ibrahim et al., 2010).

Yield of Cellulose from Agricultural Biomass

Beans peel had the highest yield at 45.5%, with white cellulose indicating high purity. Plantain peel yielded 38% and produced white cellulose, suggesting similar purity but slightly lower efficiency. Wooden wine cork had the lowest yield at 20%, with brown cellulose indicating higher lignin content and impurities (**Figure 4**). This difference in yield may be attributed to the distinct physical composition of the cellulose matrix in these plant materials (Mostafa et al., 2018). Overall, beans peel emerged as the most efficient and pure source of cellulose, making it highly suitable for bioplastic production.

FTIR Analysis

The FTIR spectroscopy results, shown in **Figure 5**, **Figure 6**, and **Figure** 7, provide detailed insights into the molecular composition of cellulose extracted from beans peel, plantain peel, and wooden wine cork. The FTIR analysis reveals a strong presence of aromatic rings, methylene (CH_2), and ether (C−O−C) groups, indicated by significant peaks at wavenumbers 898.29 cm^{-1}, 1028.75 cm^{-1}, and 1103.29 cm^{-1}, respectively. These peaks suggest the complex molecular structure of the extracted cellulose, which retains both aromatic and aliphatic characteristics (Sun et al., 2004). This finding aligns with previous studies that have identified similar functional groups in cellulose extracted from various plant sources (Hospodarova et al., 2018; Vârban et al., 2021).

Secondary amine (C−N stretch), tertiary alcohol (C−O stretch), phenol (O−H bend), and alcohol (O−H bend) groups are detected with medium peak intensities at wavenumbers 1159.20 cm^{-1}, 1203.93 cm^{-1}, 1315.75 cm^{-1}, and 1364.20 cm^{-1}, respectively. These functional groups contribute to the chemical diversity of the cellulose structure, indicating the presence of various hydroxyl and nitrogen-containing compounds (Heinz et al., 2018).
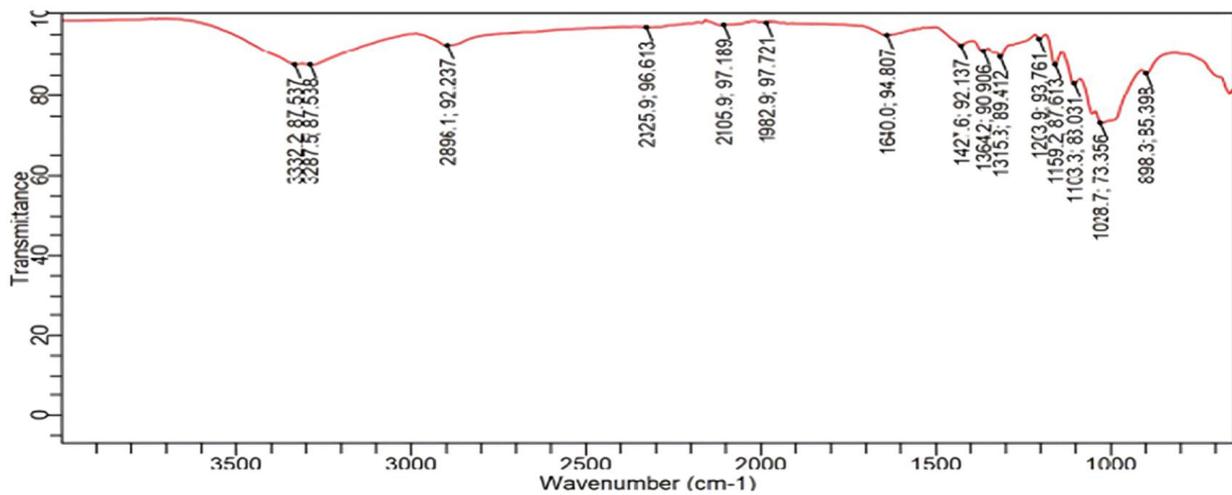
Figure 5. FTIR spectroscopy result of cellulose from beans peel (Source: Authors' own elaboration)
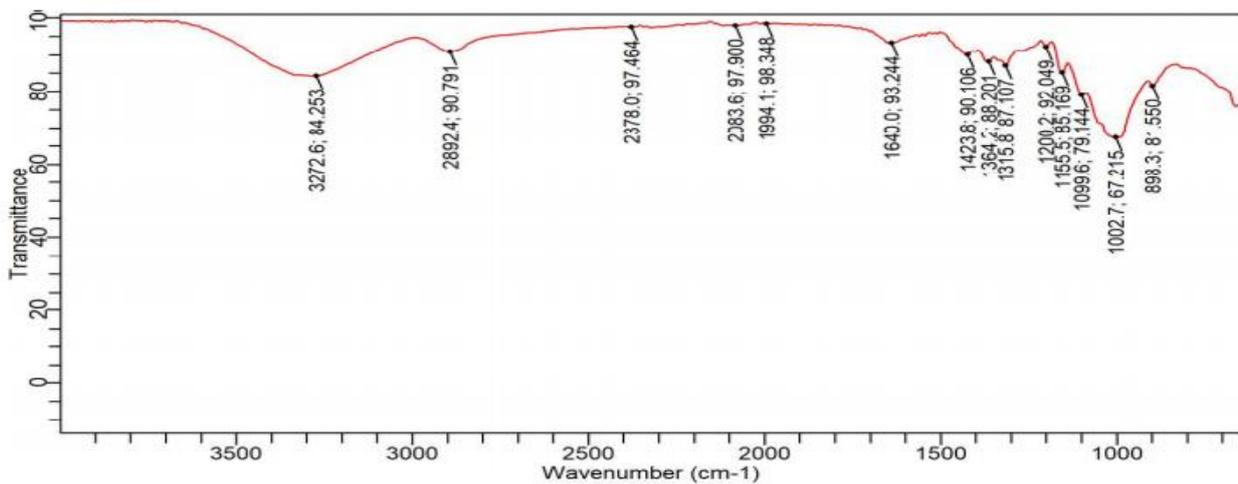

Figure 6. FTIR spectroscopy result of cellulose from plantain peel (Source: Authors' own elaboration)
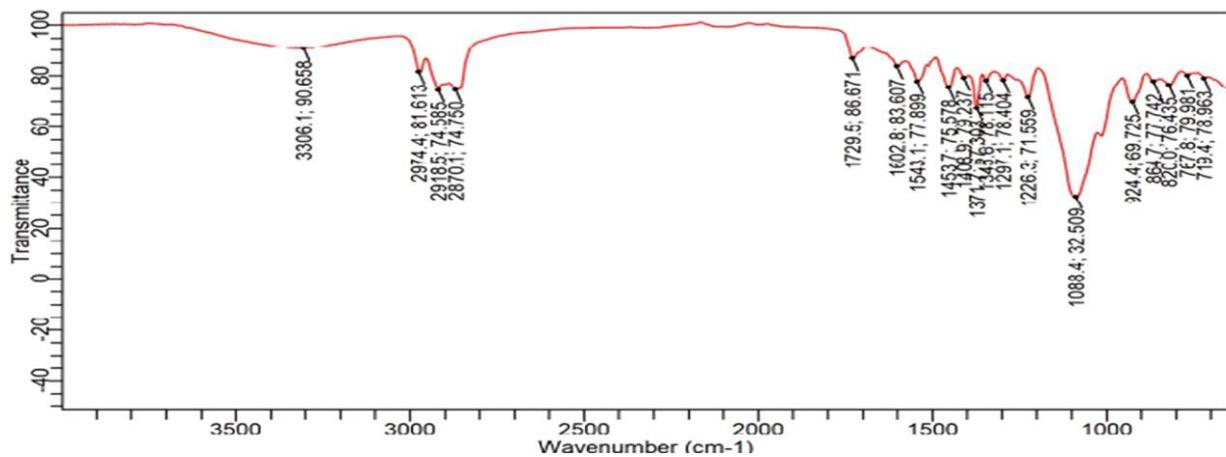

Figure 7. FTIR spectroscopy result of cellulose from wooden wine cork (Source: Authors' own elaboration)

These functional groups enhance cellulose's potential for hydrogen bonding, which contributes to its stability. The detection of these groups is consistent with the known chemical composition of cellulose, which includes a variety of hydroxyl and nitrogen-containing compounds (Hospodarova et al., 2018). Adsorbed water is identified by a medium peak at 1640.02 cm^{-1}, reflecting the hygroscopic nature of the cellulose (Carrillo et al., 2018). Additionally, weak peaks are observed for aromatic rings and alkynes at 1982.94 cm^{-1} and 2105.94 cm^{-1}, respectively, suggesting minor contributions from these groups (Vârban et al., 2021). The presence of carbon dioxide (O=C=O) is strongly indicated by a peak at 2325.85 cm^{-1}, while the methyl (C–H stretch) group shows a medium peak at 2892.14 cm^{-1}. The presence of amorphous cellulose samples may be confirmed by observing the change in the band
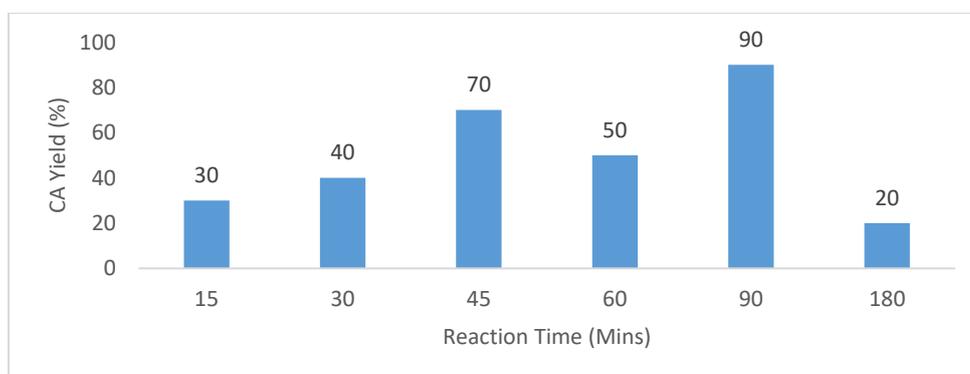
Figure 8. Cellulose acetate yield from commercial cellulose at varying time intervals (Source: Authors' own elaboration)
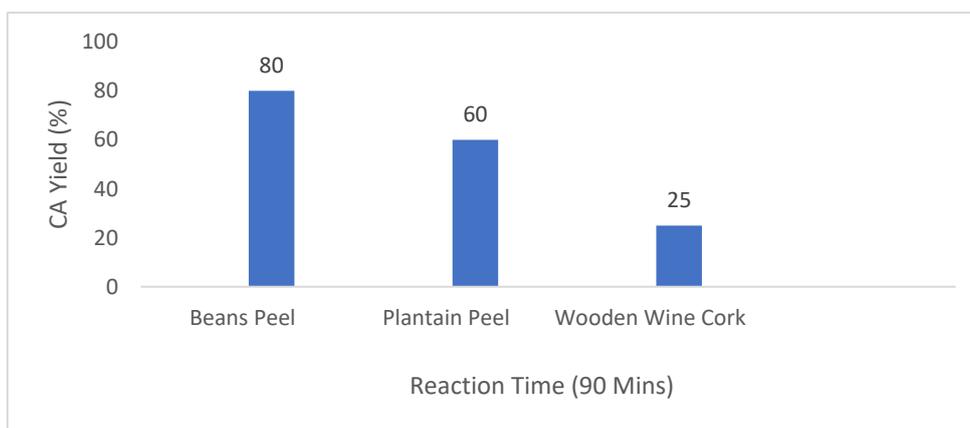

Figure 9. Cellulose acetate yield from beans peel, plantain peel, and wooden wine cork (Source: Authors' own elaboration)

at 2900 cm^{-1}, which corresponds to the stretching vibration of C–H bonds (Ciolacu et al., 2011).

Finally, the hydroxyl (O–H stretch) groups exhibit strong, broad peaks at 3287.51 cm^{-1} and 3332.24 cm^{-1}, confirming the abundant hydroxyl content, which is characteristic of cellulose and critical to its hydrogen bonding and structural integrity (Klemm et al., 2005). This is a common feature in cellulose spectra, as the hydroxyl groups play a crucial role in the material's properties. However, the FTIR spectrum of cellulose extracted from wooden wine cork, displayed in **Figure 7**, shows additional unique peaks not observed in the other samples, such as fluoro compounds (C-F stretch), peroxides (C-O-O-stretch), and nitro compounds (N-O stretch). These unique groups could be attributed to environmental factors specific to the cork source or to lignin content within the cork structure, as cork is naturally rich in lignin and other compounds absent in plant peels (Gandini et al., 2006).

Cellulose Acetate Yield

Commercial cellulose was accelerated at reaction times ranging from 30 to 180 minutes, as shown in **Figure 8**. The yield initially decreased to 30 minutes, increased steadily, and peaked at 90 minutes before declining again at 180 minutes. The highest cellulose acetate (CA) yield was observed at 90 minutes, while the lowest yield occurred at 180 minutes. This indicates the significant influence of reaction time on CA yield, with a notable increase at 90 minutes compared to other time intervals. Consequently, 90 minutes was selected as the optimal acetylation time for the plant materials due to its superior yield.

Among the materials, beans peel achieved the highest yield (80%), followed by plantain peel with a yield of 60%, and wooden wine cork with the lowest yield (25%) (**Figure 9**). The cellulose acetate from beans peel and plantain peel appeared as white powder, while that from wooden wine cork was brown, likely due to the higher lignin content. This lignin presence affected both the reaction efficiency and the characteristics of the resulting cellulose acetate. These findings are consistent with the results reported by Jassem et al. (2020).

Degree of Substitution

Beans peel exhibited the highest DS value of 2.76, indicating the most effective acetylation among the plant materials. Plantain peel followed with a lower DS value (2.22), while wooden wine cork had the lowest DS value (1.75), reflecting the least effective acetylation process. These variations highlight the differing efficiencies of acetylation across the different plant materials. This finding aligns with the results reported by Battisti et al. (2019) and Jassem et al. (2020).

Solubility Test

The results (**Table 1**) indicate that the control commercial cellulose acetate had the highest solubility in acetone (100%) and dichloromethane/acetone mixture (70%). Among the samples, plantain peel exhibited the highest solubility in acetone (60%), while beans peel showed superior solubility in dichloromethane (90%). Conversely, wooden wine cork had the lowest solubility across all solvents due to its higher lignin content and lower DS. These results suggest that higher lignin

Table 1. The solubility percentage of CA from different sources

Source of CA	Acetone %	Dichloromethane %	Dichloromethane/Acetone (1/1) %
Commercial CA	100	50	70
Beans peel	50	90	60
Plantain peel	60	30	40
Wooden wine cork	30	20	20

content and lower DS correlate with lower solubility, as also noted by Jassem et al. (2020).

Bioplastics Production

Figure 10, Figure 11 and **Figure 12** depict the biodegradable plastics produced from cellulose triacetate derived from bean peels. The sample without a plasticizer exhibited greater strength compared to those with plasticizers, suggesting that strong and durable plastics can be produced without plasticizers. However, the absence of plasticizers may limit flexibility, which could be crucial depending on the intended application (Lau et al., 2023). Furthermore, cellulose acetate derived from plantain peels proved effective for biodegradable plastic production. In contrast, cellulose acetate from wooden wine cork was unsuitable due to its low solubility and low DS, likely caused by its high lignin content (Jassem et al., 2020). These findings align with the results of Mostafa et al. (2018), who successfully produced biodegradable

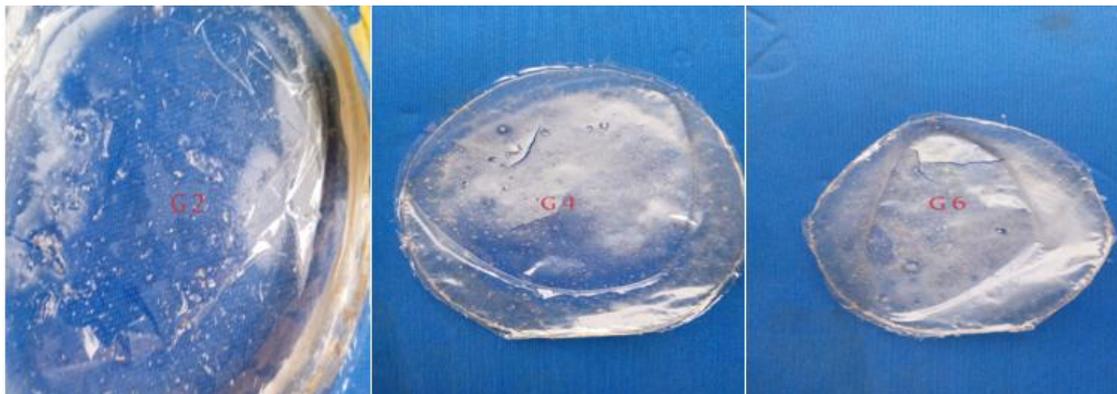**Figure 10.** Bioplastic without plasticizer (Source: Field study)

Figure 11. Bioplastics with PG2, PG4 and PG6 (PG2- 2 drops of glycerol, PG4- 4 drops of glycerol, PG6- 6 drops of glycerol) (Source: Field study)
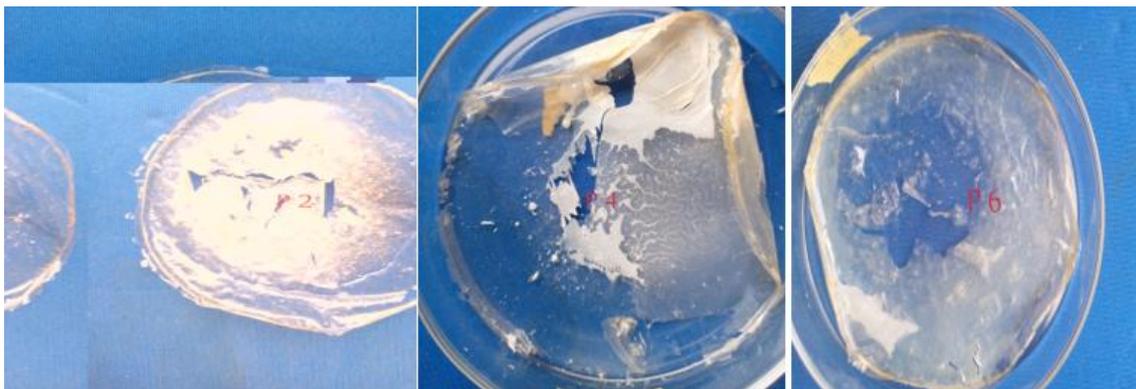

Figure 12. Bioplastics with PKO2, PKO4 and PKO6 (PKO2- 2 drops of palm kernel oil, PKO4- 4 drops of palm kernel oil, PKO6- 6 drops of palm kernel oil) (Source: Field study)
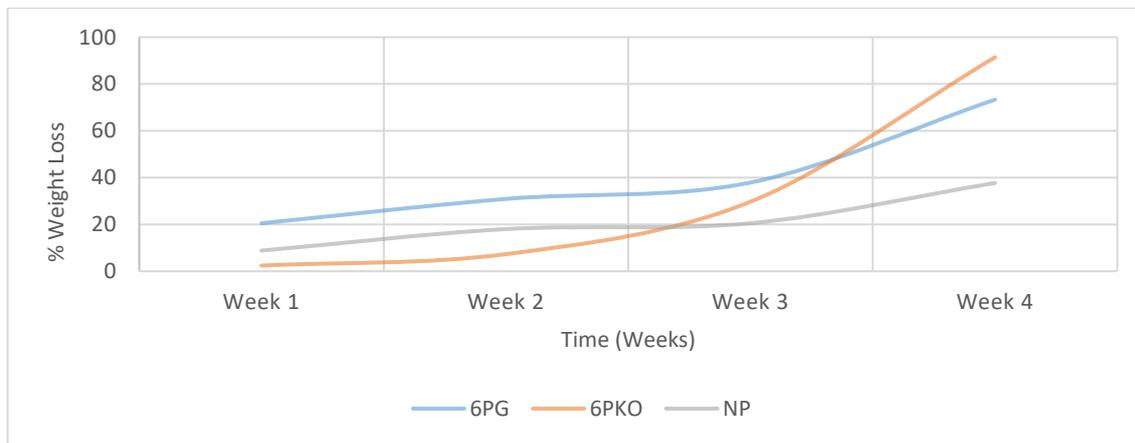
Figure 13. Biodegradability test of the bioplastics with plasticizers and no plasticizer (6PG- 6 drops of glycerol, 6PKO- 6 drops of palm kernel oil, NP- no plasticizer) (Source: Authors' own elaboration)

plastics from flax fibers, further highlighting the potential of agricultural waste for biodegradable plastic production.

Biodegradability Test of the Bioplastics

The biodegradability test results are presented in **Figure 13**. In week 1, the 6PG sample exhibited the highest degradation, followed by the NP and 6PKO samples. During week 2, the 6PG sample maintained the highest rate of degradation, again followed by the NP and 6PKO samples. In week 3, the 6PG sample continued to show the highest degradability, followed by the 6PKO and NP samples. However, by week 4, the 6PKO sample demonstrated the greatest biodegradability, followed by the 6PG sample, with the NP sample exhibiting the least biodegradability. Significant physical and chemical changes, such as polymer chain breakdown, surface erosion, and the formation of smaller fragments, were observed, confirming successful biodegradation. The data indicated that prolonged composting resulted in increased weight loss, demonstrating enhanced degradation levels over time. This outcome is consistent with the findings of Nissa et al. (2019), who observed similar degradation levels when they composted biodegradable plastics made from starch for 10 days.

CONCLUSION

This study successfully demonstrated the extraction of cellulose from agricultural waste and its conversion into biodegradable cellulose acetate. The highest cellulose yield was obtained from bean peels, which also produced the highest yield of cellulose acetate. FTIR analysis confirmed the presence of functional groups indicative of pure cellulose, while biodegradation tests validated the environmental sustainability of the produced bioplastics. These findings underscore the potential of using agricultural waste as a raw material for eco-friendly plastic production, contributing to sustainable waste management and pollution reduction.

While this study focused on three types of agricultural waste, the results provide a solid foundation for further exploration. Future research could expand on these findings by investigating a broader variety of agricultural residues and refining the extraction and conversion processes to maximize

yield and efficiency. Additionally, conducting tensile strength and elongation rate tests and comparing the results against industry standard SNI 7818: 2014 would provide a comprehensive understanding of the mechanical properties and confirm the material's suitability for its intended application.

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