# Biodegradable Polymers Based on Cellulose and Fiber from Coffee (*Coffea* spp.) and Sugarcane (*Saccharum* spp.) Residues

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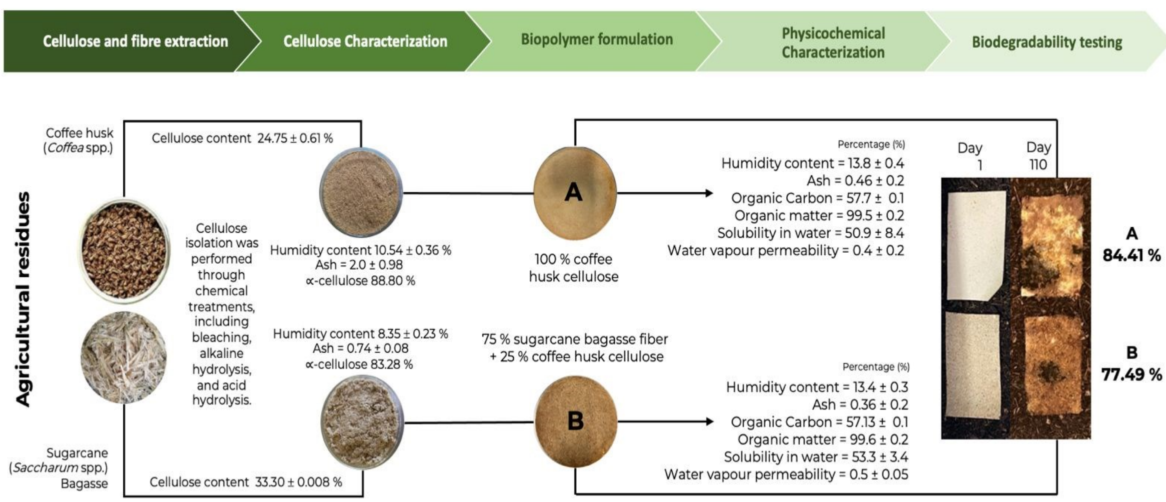
*DOI: 10.15376/biores.20.3.7856-7869*

## GRAPHICAL ABSTRACT



# Biodegradable Polymers Based on Cellulose and Fiber from Coffee (*Coffea* spp.) and Sugarcane (*Saccharum* spp.) Residues

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Agro-industrial residues, derived from cereals, fruits, and vegetables, comprise non-consumable byproducts, including stems, leaves, peels, and seeds. Globally, approximately 3,045 million tons of such material is generated annually. In Mexico, the industrial crops coffee (*Coffea* spp.) and sugarcane (*Saccharum* spp.) yield residues rich in structural components, including lignin, cellulose, and hemicellulose. This study determined the physicochemical characteristics of cellulose isolated from these residues to formulate biodegradable polymers. Cellulose isolation was performed through chemical bleaching treatments, alkaline hydrolysis, and acid hydrolysis, yielding high-purity α-cellulose at 88.8% for coffee husks and 83.3% for sugarcane bagasse, with yields of 32.8% and 29.4%, respectively. Two biopolymers were developed: (A) 100% coffee husk cellulose and (B) a composite of 75% sugarcane bagasse fiber and 25% coffee husk cellulose. Biopolymer A demonstrated superior physicochemical properties, including moisture content, water vapor permeability, and solubility. Biodegradability assessments confirmed that both biopolymers were compostable within 110 days, exhibiting degradation extents of 84.4% (A) and 77.5% (B), primarily converting into organic matter and $CO_2$. These findings indicate that coffee and sugarcane agro-industrial residues are viable feedstocks for sustainable biopolymer production.

*DOI: 10.15376/biores.20.3.7856-7869*

*Keywords: Agricultural residues; Cellulose; Coffee husks; Sugarcane bagasse; Biodegradability*

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## INTRODUCTION

Global plastics production increased from 245 million tons in 2008 to 335 million tons in 2016 and is estimated to reach one billion tons by 2050 (FAO 2011; Kumar *et al*. 2017). The most produced plastics worldwide include polypropylene (19%), low- and high-density polyethylene (17% and 12%, respectively), polyvinyl chloride (11%), polystyrenes (7.5%), polyurethanes, and polyethylene terephthalate (6.5%). In Mexico, the plastics industry has grown annually 3.03%, exceeding the country's average economic growth (2.92%) between 2003 and 2013 (Pérez 2014). In 2015, the per capita generation of plastic waste was 1.2 kg per day, equivalent to 53.1 million tons per year. Because these materials take between 150 and 500 years to degrade, their accumulation has led to the widespread presence of microplastics in various ecosystems (Sotelo *et al*.

2019). One solution to the problems associated with synthetic plastics is the use of bioplastics, which are biodegradable polymers made from biomass sources such as starch, fiber, and cellulose (Azmin *et al.* 2020). Cellulose and its byproducts could replace synthetic plastics in some applications, including food packaging, personal care products, textiles, and other pharmaceutical items, if they exhibit adequate biodegradability, low toxicity and can be developed at a low cost (Liu *et al*. 2021; Chandel *et al*. 2023; Nocca *et al*. 2023).

Coffee (*Coffea* spp.) and sugarcane (*Saccharum* spp.) are crops of great economic importance in Mexico, particularly in the state of Veracruz, where a significant portion of their production is concentrated. These crops contribute to the local and national economy and generate large amounts of agro-industrial waste, such as coffee husks and sugarcane bagasse. These residues, which are not intended for human consumption, represent a valuable source of structural components, including lignin, cellulose, and hemicellulose, making them suitable to produce biodegradable polymers (Ruilova and Hernández 2014; Arias and Meneses 2016). Reusing this material reduces the environmental impact associated with improper disposal and offers a sustainable alternative for the materials industry, promoting a circular economy in the agricultural sector (Ballinas-Casarrubias *et al*. 2016; García *et al*. 2020).

Cellulose- or fiber-derived polymers are more biodegradable than various synthetic plastics, including others of biological origin and some cellulose derivatives (Hubbe *et al*. 2025). Furthermore, their use contributes to reducing fossil fuel consumption and to the mitigation of both $CO_2$ emissions and plastic pollution in the environment (Rosenboom *et al*. 2022). Additionally, research has demonstrated improvements in the physicochemical properties of these materials through the combination of cellulose and fiber in their production (Azmin *et al*. 2020). However, the properties of these materials must continue to be tested and examined for the proper development of applications with high potential to replace traditional plastics, as has been done for cellulose derivatives such as cellulose acetate, nanocellulose, cellulose sulfate, methylcellulose, and carboxymethylcellulose, to mention some of the most promising in the food industry (Nath *et al*. 2024). In this context, the present study focuses on the isolation and characterization of cellulose from coffee (*Coffea* spp.) and sugarcane (*Saccharum* spp.) residues, to develop biodegradable polymers as eco-friendly alternatives and analyze their biodegradation.

## EXPERIMENTAL

### Sample Collection

Samples of coffee husks (*Coffea* spp.) were collected from the Acatepec Coffee Mill in Huatusco, Veracruz, Mexico. Sugarcane bagasse (*Saccharum* spp.) samples were obtained from the Ingenio Grupo Azucarero San Pedro S.A. de C.V. in Saltabarranca, Veracruz, Mexico (Fig. 1).

### Fiber Delignification and Cellulose Isolation

Sugarcane bagasse fiber extraction was carried out according to the method reported by Azmin *et al.* (2020), which involved mixing and stirring the ground bagasse with NaOH for 60 min, followed by washing and filtering until the pH was neutralized. After drying the extracted fiber at 50 °C for 24 h, it was subjected to bleaching using a solution of acetic acid ($CH_3COOH$) and sodium chloride (NaCl). A second filtering and washing was performed to re-neutralize. Finally, the bleached fiber was also dried under the conditions described above.


**Fig. 1.** Samples of coffee husks (A) and sugarcane bagasse (B)

Cellulose isolation was performed according to the methods proposed by Cazaurang *et al.* (1990) and Ortega (2017). For coffee husks, an alkaline treatment with sodium hydroxide (NaOH) was applied for 60 min, followed by bleaching with sodium chlorite ($NaClO_2$) and acid hydrolysis with sulfuric acid ($H_2SO_4$). Due to its fibrous structure, two cycles of alkaline hydrolysis and bleaching were performed for sugarcane bagasse.

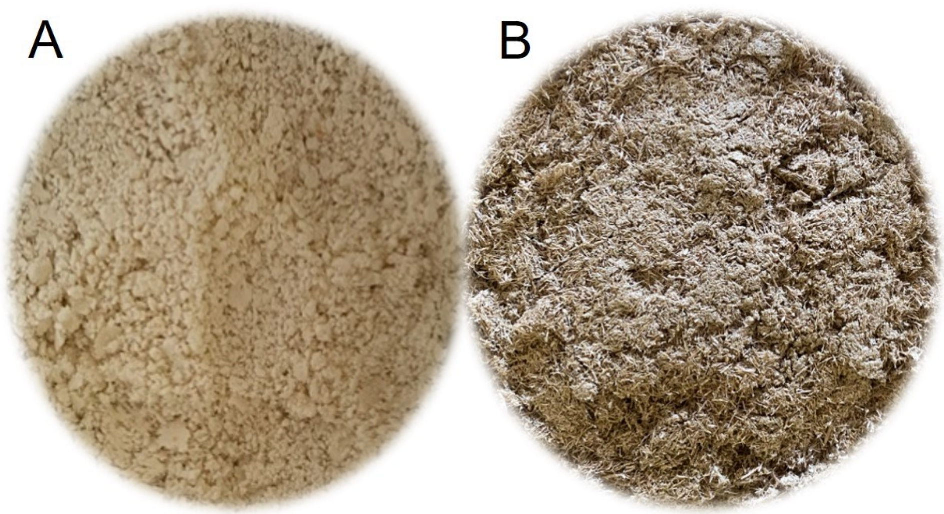


**Fig. 2.** Coffee husk cellulose (A) and sugarcane bagasse fiber (B)

*Characterization of cellulose*

The moisture content was determined using the AOAC method 925.10 (2023), which involves drying 2 g of ground samples in an air oven at 130 °C for 1 hour. Ash content was determined by muffle furnace incineration at 550 °C, following AOAC 923.03 (2023). Likewise, α-cellulose was determined as the portion of crude cellulose that is insoluble in 17.5% NaOH, following the TAPPI method T203 m-58 (2022). The cellulose yield (*CY*) was calculated using Eq. 1,

$$CY\,(\%) = (c/z) \times 100 \qquad (1)$$

where $c$ is the amount of cellulose extracted (g), and $z$ is the initial weight (g) of the sample.

## Biopolymer Formulation

Two biopolymers were formulated: (A) 100% coffee husk cellulose and (B) a composite of 75% sugarcane bagasse fiber and 25% coffee husk cellulose (Fig. 3). Both formulations were selected in preliminary tests based on physical characteristics such as morphology and texture that allowed their manipulation for the production of biopolymeric sheets, as well as some of the physicochemical properties described in the following section.

For each formulation (A and B), 1.5 g of material was weighed and mixed with 1 mL of glycerol, 40 mL of distilled water, and 0.5 g of sorbitol. Additional distilled water was added at a 1:10 ratio (w/v), and the mixture was subjected to continuous agitation with heating until complete evaporation, achieving a homogeneous viscous consistency. The resulting mixture was transferred to a glass Petri dish and evenly spread across the surface for drying at 50 °C for 4 h in a Felissa MCA oven. Subsequently, biopolymers A and B were maintained at room temperature (~25 °C) for approximately 72 h until reaching constant weight.

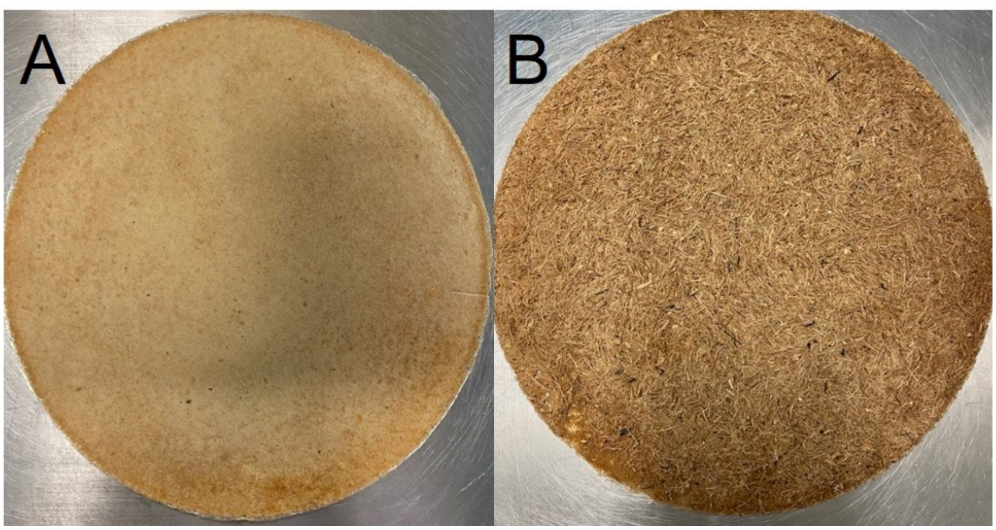


**Fig. 3.** Formulated biopolymers (A: 100% coffee husk cellulose; B: 75% sugarcane bagasse fiber and 25% coffee husk cellulose)

## Characterization of Biopolymers

The formulated biopolymer sheets (A and B) were characterized in terms of variables associated with their functionality and stability, to establish the parameters required for biodegradation testing.

*pH, ash, and moisture content*

The pH was measured using the potentiometric method AOAC 943.02 (2023). A 1 g sample was taken and homogenized in 10 mL of deionized water. The mixture was allowed to stand for 30 min, and the pH was measured using a potentiometer at 20 °C.

Ash and moisture content were determined by gravimetric analysis described in the previous section, following the AOAC 923.03 (2023) and AOAC 925.10 (2023) methods, respectively.

*Water vapor transmission and permeability*

Water vapor transmission (*Wvt*) was assessed following the ASTM E96-22 (2022) method. The biopolymers were placed in volumetric containers with distilled water and positioned in a desiccator containing desiccant. Specimen weights were recorded at 24-h intervals over a 9-day period (Chariguamán 2015). The driving force for water vapor transmission was derived from the vapor pressure differential between the test cup's interior (100% RH) and exterior (100% RH), expressed in kPa. Water vapor transmission rate and permeability coefficients were calculated using Eqs. 2, 3, and 4,

$$Wvt = \Delta m/(t \times A) \quad (2)$$

where *Wvt* is the degree of water vapor transmission ($g \cdot m^{-2} \cdot h^{-1}$), $\Delta m$ is the mass change occurring over time (g), $t$ is the time between readings (h), and $A$ is the sample test area ($m^2$). The water vapor permeability is given by Eq. 3,

$$Wvp = (\Delta m \times \varepsilon) / (t \times A \times \Delta P) \quad (3)$$

where *Wvp* is the water vapor permeability ($mm \cdot g \cdot kPa^{-1} \cdot m^{-2} \cdot h^{-1}$), $\Delta m$ is the mass change occurring over time (g), $\varepsilon$ is the film thickness (mm), $t$ is the time between readings (h), $A$ is the sample test area ($m^2$), and $\Delta P$ is the vapor pressure difference (kPa). The vapor pressure difference is given by Eq. 4,

$$\Delta P = S \times (R_1 - R_2) \quad (4)$$

where $\Delta P$ is the vapor pressure difference (kPa), $S$ is the water saturation pressure at test temperature (kPa), $R_1$ is the relative humidity of the part with the highest vapor pressure (%), and $R_2$ is the relative humidity of the part with the lowest vapor pressure (%).

*Water solubility*

Solubility was determined as the percentage of water-soluble biopolymer, following the method described by Flores (2016). The procedure involved immersing the biopolymer in distilled water at room temperature (25 ± 2 °C) for 24 h under static conditions. The insoluble fraction was then filtered and oven-dried. Initial ($W_0$) and final dry weights ($W_1$) were recorded after 24 h of drying at 105 °C. Water solubility (*WS*) was calculated using Eq. 5,

$$WS\ (\%) = ((W_0 - W_1) / W_0) \times 100 \quad (5)$$

*Organic carbon and matter*

Organic carbon (%) and organic matter (%) were quantified according to Galindo (2017) using the following equations. The organic matter (*OM*) percentage was calculated from the previously determined ash content using Eq. 6:

$$OM\ (\%) = 100 - Ash\ (\%) \quad (6)$$

The organic carbon (*OC*) percentage was calculated using Van Bemmelen's conversion factor of 1.724 in Eq. 7:

$$OC\ (\%) = OM\ (\%) / 1.724 \quad (7)$$

*Biodegradability test*

Following the ASTM D5988-12 (2012) and Reyes-Samilpa *et al.* (2020) methodologies, biodegradability was evaluated using a 40 L airtight container with a glass lid. Biopolymer samples (350 $mm^2$) were vertically arranged in 7,429.2 g of rock-free worm humus substrate alongside an UNE-EN ISO 14855-2 (2019) compliant commercial compostable control. Environmental conditions were maintained at 25 ± 0.3 °C and 99% RH using 1,061 mL distilled water, monitored with an MCA Beurer thermohygrometer (± 0.5 °C, ± 2 to 3% RH accuracy). Aeration cycles consisted of 30-min exposures every 72 h (days 0 - 20) and every 96 h (days 21 - 110), continuing for 110 days. Initial sample thicknesses and dry weights were recorded prior to testing. Mass loss (*ML*) was calculated using Eq. 8,

$$ML\ (\%) = W_1 - (W_2/W_1) \times 100 \quad (8)$$

where $W_1$ is the starting weight (g) and $W_2$ is the final weight after 110 days (g).

## Statistical Analysis

The moisture content, water vapor permeability, and solubility of biopolymer formulations (A: 100% coffee husk cellulose; B: 75% sugarcane bagasse fiber/25% cellulose) were analyzed using small-sample statistical inference methods with confidence intervals. A single-factor analysis of variance (ANOVA) was performed using statistical software (Minitab, Minitab LLC, version 18.1, State College, PA, USA) to compare the two formulations, with the null hypothesis evaluated through p-value analysis at a 0.05 significance level to determine statistically significant differences in material properties.

# RESULTS AND DISCUSSION

## Characterization of Agro-industrial Waste

The moisture content of coffee husks (10.54%) fell within the 8.6 to 15% range reported by Arias and Meneses (2016) and Suárez (2018), while sugarcane bagasse showed lower moisture (8.35%; Table 1) than values reported by Flores (2016) and Ruilova and Hernández (2014). The cellulose content in coffee husks (24.8%) was lower than the literature values (Collazo-Bigliardi *et al.* 2018), including Arias and Meneses (2016). In contrast, sugarcane bagasse cellulose (33.3%) was significantly lower than the reported values by Ruilova and Hernández (2014) (44.9%) and Flores (2016) (56.8%).

**Table 1.** Physicochemical Characterization of Agro-industrial Residues: Coffee Husks (*Coffea* spp.) and Sugarcane Bagasse (*Saccharum* spp.)

| Agro-industrial residue | Humidity content (%) | Cellulose content (%) | Cellulose composition (%) | |
|---|---|---|---|---|
| Sugarcane bagasse (*Saccharum* spp.) | 8.35 ± 0.23 | 33.30 ± 0.008 | Humidity content | 4.16 ± 1.42 |
| | | | α-cellulose content | 83.28 |
| | | | Ash content | 0.74 ± 0.08 |
| Coffee husks (*Coffea* spp.) | 10.54 ± 0.36 | 24.75 ± 0.61 | Humidity content | 3.62 ± 1.23 |
| | | | α-cellulose content | 88.80 |
| | | | Ash content | 2.0 ± 0.98 |

The cellulosic moisture of sugarcane bagasse (Table 1) was lower (4.16%) than that reported in the literature (Flores 2016). This fact is important because cellulose is an inert compound and its application as an additive element is limited to moisture retention (Barros 2009). Low humidity reduces the possibility of microbial growth and its rapid metabolic activity prevents deterioration and physical-chemical changes (Borah *et al*. 2019; Azmin *et al*. 2020).

The sugarcane bagasse cellulose exhibited low ash content (0.74%), within the range reported by Flores (2016). According to Rowell (2012), lower ash values indicate higher cellulose purity, reflecting the effective removal of inorganic components (mineral salts, silica, and metals) during isolation, which otherwise impair cellulose properties (strength, whiteness, processability). In contrast, coffee husks showed higher ash content (2.0%) compared to values reported by Aquino *et al.* (2012) (0.49%) and Collazo-Bigliardi *et al*. (2018) (0.19%). The ash was attributed by these authors to residual mineral content influenced by plant species, soil conditions, and cultivation practices, which also affects residue longevity. The α-cellulose content was 83.3% for sugarcane bagasse and 88.8% for coffee husks, representing the purest cellulose fraction and serving as a key quality indicator for industrial applications (Rowell 2012). Pandey *et al.* (2000) report that sugarcane bagasse constitutes a promising α-cellulose source, with post-treatment content exceeding 50%, making it suitable for industrial use. While coffee husks demonstrate lower α-cellulose content than other agricultural residues, they remain a viable cellulose source, particularly for applications requiring less stringent purity (Murthy and Madhava 2012).

## Physicochemical Characterization of Biopolymers

The observed humidity variations (Table 2) result from the hygroscopic nature of cellulosic materials, which gradually absorb or release moisture until reaching equilibrium with the environment (Wang *et al*. 2018). Formulation B exhibited lower moisture content (13.5%), which was attributable to its reduced cellulose and fiber composition (Table 10). This lower moisture content inhibits mold growth, which could compromise biopolymer properties. Notably, both formulations (A and B) demonstrated moisture levels below literature-reported values (Azmin *et al*. 2020), with no statistically significant differences between them ($p > 0.05$). Ash content analysis provides an estimate of the material's inorganic load. Table 3 reveals that organic substances predominated over inorganic components in both biopolymers. These values indicate the proportion of material potentially biodegradable in compostable media (García 2020), with higher organic content corresponding to greater biodegradation potential.

**Table 2.** Physicochemical Characteristics of the Formulated Biopolymers

| BP | pH | Percentage (%) | | | | | Water Vapor Permeability (mm·g·kPa$^{-1}$·m$^{-2}$·h$^{-1}$) |
|---|---|---|---|---|---|---|---|
| | | Humidity content | Ash | Carbon Organic | Organic Matter | Solubility in Water | |
| A | 7 | 13.848 ± 0.406 | 0.435 ± 0.246 | 57.751 ± 0.142 | 99.56 ± 0.246 | 50.915 ± 8.499 | 0.479 ± 0.202 |
| B | 7 | 13.460 ± 0.381 | 0.361 ± 0.232 | 57.794 ± 0.134 | 99.638 ± 0.232 | 53.354 ± 3.443 | 0.664 ± 0.050 |

BP: Formulated biopolymers (A: 100% coffee husk cellulose; B: 75% sugarcane bagasse fiber and 25% coffee husk cellulose)

The organic carbon and matter content in biopolymers A and B serve as key indicators of their biodegradability and sustainability, reflecting their natural-derived composition (Rowell 2012). Both formulations demonstrate high organic carbon content, making them ideal raw materials for biodegradable polymers that combine mechanical performance with degradation capacity (Pandey *et al*. 2000), particularly for applications requiring strength-degradability balance (Murthy and Madhawa 2012). Water solubility analysis revealed no significant differences between formulations ($p > 0.05$). Due to its insoluble nature, solubility is inversely proportional to cellulose concentration due to its insoluble nature. This relationship determines the biopolymers' suitability for specific applications (Morales 2014; Flores 2016).

Consistent with Moreno's findings (2015), optimal biofilms should maintain limited water absorption to preserve impermeability and insolubility. While literature reports solubility ranges of 9 to 35% in cassava starch films (Paspuel 2016) and 16 to 38% in coffee-corn starch bio sheets (Toala and Sarmiento 2019), the present sugarcane bagasse cellulose formulations showed higher solubility (48.0 to 53.2%) (Flores 2016), classifying them as moderately soluble materials. This property enables diverse applications, including edible coatings, biodegradable packaging, and pharmaceutical/textile uses where moderate moisture barriers are required (Chariguamán 2015; Paspuel 2016; Toala and Sarmiento 2019).

Water vapor permeability (*Wvp*) analysis showed no statistically significant differences between formulations ($p > 0.05$). Biopolymer A demonstrated superior barrier properties (0.479 g·mm·kPa$^{-1}$·m$^{-2}$·h$^{-1}$), which is attributable to its higher cellulose content and consequent hydrophobicity (Majeed *et al*. 2013), while biopolymer B exhibited greater vapor transmission (0.664 g·mm·kPa$^{-1}$·m$^{-2}$·h$^{-1}$).

## Biodegradability

Table 3 presents the mean mass loss in the formulated biopolymers. During the 110-day experiment, formulations A and B exhibited mass losses of 84.4% and 77.5%, respectively, surpassing the 60% biodegradability threshold specified in ASTM D5988-12 (2012). In contrast, the control sample (C) exhibited no structural changes or measurable mass loss, despite the manufacturer's claims of 60 to 180-day biodegradability, which is compliant with the UNE-EN ISO 14855-2 (2019) standard.

**Table 3.** Mass Losses in the Biopolymers Over 110 Days of Testing

| BP | Mass* (g) | |
|---|---|---|
| | Initial | Final |
| A | 1.9000 ± 0.1611 | 0.2968 ± 0.0910 |
| B | 2.0346 ± 0.1529 | 0.4578 ± 0.0801 |
| C (Control) | 0.0636 ± 0.0069 | 0.0636 ± 0.0069 |

BP: Formulated biopolymers (A: 100% coffee husk cellulose; B: 75% sugarcane bagasse fiber and 25% coffee husk cellulose). C: commercial compostable bag. *Mean ± SD (n =5).

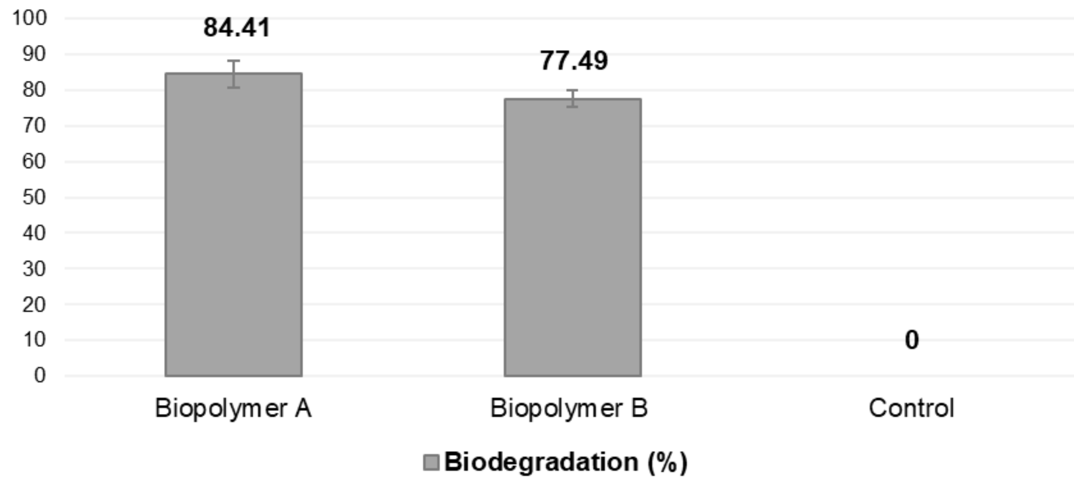


**Fig. 4.** Biodegradability percentages of biopolymers over 110 days of testing (mean ± SD; n =5)

The formulated biopolymers demonstrated significant biodegradation, with biopolymer A degrading at 84% and biopolymer B at 77% over the 110-day (15-week) experimental period (Fig. 4). These results compare favorably with literature values: Toala and Sarmiento (2019) reported 60% degradation for coffee husk (*Coffea arabica*) corn starch (*Zea mays*) composites within 6 weeks (42 days), while Meza (2016) achieved 80% degradation for potato waste-based bioplastics in 10 weeks (70 days). All cases, including the present study, meet the biodegradation requirements specified in the UNE-EN ISO 14855-2 (2019) rule.

For cellulose production, the valorization of agro-industrial wastes—specifically coffee husks (*Coffea* spp.) and sugarcane bagasse (*Saccharum* spp.)—represents a strategically important approach that addresses both environmental sustainability and economic viability. This methodology directly aligns with circular economy principles by (1) transforming agricultural byproducts into high-value materials, (2) minimizing waste generation through complete resource utilization, and (3) creating sustainable alternatives to conventional plastics while maintaining competitive production costs. The demonstrated technical feasibility of converting these residues into functional biopolymers with desirable physicochemical properties establishes the need to evaluate other formulations in future work, as well as to include processes that optimize economic resources while ensuring a low environmental impact, for example, with the application of the complete kraft process for the elimination of lignin in the agro-industrial residues studied.

## CONCLUSIONS

1. This study demonstrated that coffee husks (*Coffea* spp.; 10.5% moisture, 24.7% cellulose) and sugarcane bagasse (*Saccharum* spp.; 8.3% moisture, 33.3% cellulose) can serve as effective feedstocks for high-purity cellulose isolation, yielding α-cellulose contents of 88.8% and 83.2%, respectively, through optimized chemical treatments (bleaching/alkaline/acid hydrolysis).
2. While cellulose yields were moderately lower than some reported in the literature, the exceptional purity validates the efficacy of the isolation protocol. It confirms these agro-industrial residues as viable raw materials for developing biodegradable polymers that meet industrial performance standards.
3. The formulated biopolymers—A (100% coffee husk cellulose) and B (75% sugarcane bagasse fiber/25% coffee husk cellulose)—exhibited advantageous physicochemical properties. Biopolymer A demonstrated superior performance in terms of moisture content (13.5%), water vapor permeability (0.479 $g \cdot mm \cdot kPa^{-1} \cdot m^{-2} \cdot h^{-1}$), and solubility, rendering it particularly suitable for applications that demand enhanced barrier properties.
4. Both formulations showed minimal ash content (A: 2.0%; B: 0.74%), confirming high cellulose purity and low inorganic contamination. Their substantial organic carbon and matter content further validated their industrial applicability and biodegradation potential. These characteristics collectively position these materials as competitive alternatives for sustainable packaging and other applications that require a balance of mechanical and barrier properties.
5. The biodegradability test indicated that, under controlled conditions of temperature and relative humidity, the formulated biopolymers were compostable in less than four months (110 days), biodegrading up to 84.41% (A) and 77.49% (B), with the majority of the mass of both formulations being converted into organic matter and $CO_2$.
6. The use of agro-industrial waste, such as coffee husks (*Coffea* spp.) and sugarcane bagasse (*Saccharum* spp.), to produce biopolymers is significant from both environmental and economic perspectives. This approach is aligned with the principles of the circular economy, which aims to minimize waste, maximize resource efficiency, and promote sustainable development.

## ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support provided by Tecnológico Nacional de México (Project No. 10459.21-P).

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Article submitted: April 28, 2025; Peer review completed: June 30, 2025; Revised version received and accepted: July 20, 2025; Published: July 31, 2025.
DOI: 10.15376/biores.20.3.7856-7869