Original Paper

Cocoa shell bioplastic: a circular path towards sustainability

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Received: 1 September 2024 | **Accepted:** 27 October 2024 | **Published:** 9 November 2024

Keywords: bioplastic synthesis; agricultural waste; sustainability; circular economy; biodegradability.

Abstract. *This study explored the potential of cocoa shells as a sustainable feedstock for bioplastic production. Two treatments, T1 and T2, using a mixture of cocoa shell flour and cassava starch, were evaluated based on their filtration, solubility, elongation, strength, moisture content, and biodegradability. The results showed that the cocoa shell flour had favorable* *rheological and thermal properties (viscosity: 39 mPa s; gelatinization temperature: 80°C), though it had high moisture (24.94%) and ash content (23.93%). Treatment T2 demonstrated superior filtration and solubility, while T1 excelled in elongation, strength, and biodegradability. Both treatments showed good solubility, indicating potential compatibility with different solvents. These findings suggest that cocoa shells are a promising and eco-friendly resource for bioplastic production.*

1. Introduction

Agro-food waste, a significant by-product of agribusiness, holds immense untapped potential for resource recovery and sustainable innovation. As noted by Matei et al. (2021), this waste stream offers valuable opportunities across various sectors. While traditionally used primarily as animal feed, recent studies, such as those by Caliceti et al. (2022), have demonstrated its wider applicability in multiple industries. Agro-food by-products like peels, pomace, seeds, and leaves are rich in bioactive compounds, including phenols, anthocyanins, peptides, and fatty acids. These by-products also contain valuable fibers and enzymes, making them ideal for applications in functional foods, pharmaceuticals, and cosmetics (Del Río et al., 2021). This combination of bioactive components and structural elements positions agro-food waste as a promising and versatile raw material for diverse industrial uses (Atiwesh et al., 2021).

The cocoa industry exemplifies the significant economic and environmental losses caused by inefficient management of agri-food waste (Torres, 2023). Parra et al. (2018) emphasize the vast amounts of waste produced in this sector, representing a missed opportunity for resource recovery. In Ecuador alone, around 700,000 tons of cocoa waste are generated annually, with only 10% of the cocoa fruit being utilized (Romero, 2020). Key by-products, such as mucilage, which is rich in pectin, and cocoa pods, are frequently discarded in large quantities, contributing to soil degradation and increasing the risk of disease (Plasencia et al., 2021). This highlights the need for more sustainable practices in the cocoa industry to capitalize on these underutilized resources.

The environmental challenges posed by plastic waste underscore the urgent need for sustainable waste management solutions. The widespread production of conventional plastics has caused severe environmental harm, with vast amounts of plastic waste accumulating and disrupting ecosystems and wildlife. Globally, around 100 million tons of plastic are produced each year, with 25 million tons consisting of unnecessary items that directly contribute to pollution (Barrientos, 2019; Ospina, 2019). This highlights the critical importance of reducing plastic waste and adopting eco-friendlier alternatives to mitigate further environmental damage.

Bioplastics, made from plant-based materials, provide a sustainable alternative to conventional plastics, offering significant environmental benefits and with significant potential for recovery of bio-based plastics at end-of-life (Ritzen et al., 2023). Their biodegradable nature helps address the growing issue of agri-food waste, particularly in the cocoa industry. By utilizing cocoa by-products for bioplastic production, we can reduce dependence on traditional plastics, lessen environmental harm, and promote more sustainable practices. Bioplastics have the potential to lower the environmental footprint of plastic production and disposal by decreasing greenhouse gas emissions, reducing reliance on fossil fuels, and encouraging greener industrial processes. Moreover, bioplastics can play an important role in building resilient infrastructure and fostering innovation. The development of bioplastics from renewable resources marks a vital step toward resource-efficient technologies that drive sustainability and promote a circular economy (Rosenboom et al., 2022).

Agricultural waste is increasingly recognized as an ideal feedstock for bioplastic production due to its high content of cellulose, pectin, and starch, which make it highly suitable for conversion into bioplastics. This approach not only addresses waste disposal challenges but also mitigates its negative effects on soil and water resources (Choudhary et al., 2024). The present study focuses on the utilization of cocoa shells for bioplastic synthesis, beginning with a detailed characterization of their properties. This analysis will guide the development of precise experimental procedures to create a safe, sustainable bioplastic by-product, contributing to environmental sustainability and advancing a more efficient circular economy.

This study adds to the scientific community by providing a new avenue for bioplastic development from cocoa shells. It also offers practical insights into formulating bioplastics with enhanced properties by mixing cocoa shell flour with cassava starch. Such research helps advance the field of green materials science, particularly in terms of resource utilization and sets the stage for further research into improving and scaling bioplastic formulations for commercial applications.

2. Methodology

The research was conducted in three sequential phases: first, the extraction of cocoa shell flour; second, the development of the bioplastic; and third, the statistical analysis of the material's resulting properties. Each phase was designed to systematically assess the potential of cocoa shell flour as a viable feedstock for bioplastic production. This study adapted several methods from prior research to align with its objectives. Notably, the work of Jõgi & Bhat (2020), which outlines the direct extraction of bioplastics from biomass, informed this investigation development. For sample collection and pre-treatment, the methodology followed Azmin et al. (2020) and other relevant studies, as detailed below.

2.1. Obtaining cocoa shell flour

Organic cocoa shells, specifically from fine aroma Nacional cocoa, were sourced as raw material from the Fortaleza del Valle Association, a renowned farmers' collective in Calceta, Manabí Province, Ecuador, known for its commitment to organic farming. The cocoa shells used for flour production were directly obtained from this association, ensuring high-quality, organically grown materials.

Flour extraction followed the wet method described by Herrera et al. (2020). Three kilograms of cocoa shells were initially reduced in size and ground to form a liquid suspension. This suspension was then filtered and allowed to settle for 24 hours to isolate the flour.

Physicochemical characterization was performed according to Cando (2021), assessing key parameters such as density, viscosity, pH, gelatinization temperature, moisture content, dry matter, water absorption index, water solubility index, swelling power, and ash content, the latter determined by calcination.

2.2. Bioplastics production process

Bioplastics were synthesized using a mixture of 2 g of cocoa shell flour and cassava starch, following a completely randomized experimental design. Building on the methodology of Lizinka (2022), two levels of cassava starch (5% and 10%)

and three stirring times (5, 10, and 15 minutes) were evaluated. The bioplastics were prepared using the casting technique, where 10 g of each solid were mixed in 100 ml of distilled water. The mixture was stirred at 500 rpm for the specified times and then gelatinized at 70°C for 30 minutes. To enhance flexibility, 3 ml of glycerin and 1 ml of acetic acid were added as plasticizers, resulting in a viscous solution that was molded into bioplastic sheets.

The bioplastic mixture was poured into Petri dishes and dried in an oven at 35°C for 24 hours. Subsequent analyses, including tests for tensile strength, water filtration (resistance), elongation, and solubility, were performed in accordance with the methods outlined by Cedeño & Gilces (2022).

Moisture content was measured by first weighing the sample, then drying it in an oven at 105°C for 24 hours and reweighing it. This process was repeated until a constant weight was achieved. The moisture content was calculated using the method described by Azmin et al. (2020):

$$Moisture(\%) = \frac{(w - w_t)}{w} * 100$$

where w is the initial weight of the bioplastic, and w_t is the final weight or constant dry weight of the bioplastic.

To assess biodegradability, bioplastic films with an initial weight (W_0) were buried at a depth of 3.5 cm in moist humus soil within a glass container. The samples were retrieved after 2, 4, 6, 8, and 10 days, cleaned of any adhering soil particles, and dried at 105°C for 3 hours to determine their final weight (Wt). The percentage of weight loss (%WL) was then calculated using the method outlined by Sernaqué et al. (2020):

$$WL(\%) = \frac{(W_0 - W_t)}{W_0} * 100$$

2.3. Statistical analysis

To evaluate whether significant differences existed between treatment means, an ANOVA was conducted. Post-hoc tests were employed to identify specific mean differences. Before analysis, the assumptions of normality and homogeneity of variance for the ANOVA were assessed using the Shapiro-Wilk test. In instances of non-normality, the non-parametric Kruskal-Wallis test was utilized. To examine correlations between variables, Pearson correlation coefficients were calculated for normally distributed data, while Spearman correlation coefficients were used for non-normally distributed data.

3. Results and discussion

3.1. Characterization of cocoa shell-based flour

The process of obtaining cocoa shell flour from 7 kg of fine aroma domestic cocoa resulted in 700 g of flour through wet extraction, yielding 10%. In contrast, Muñoz et al. (2020) reported a higher yield of 29.08% using a drying and grinding method for cocoa shells. The disparity in yields may be attributed to differences in processing techniques and material conditions. Additionally, the chemical analysis conducted by Muñoz et al. (2020) revealed a crude fiber content of 26.75% and carbohydrates comprising 52.61%, highlighting the potential of cocoa shells as a valuable agro-industrial by-product.

The pH measurement yielded a result of 8.13, indicating that the product derived from the cocoa shell is slightly alkaline, which suggests areas for improvement. In contrast, Guamán's study (2022) on bioplastics made from triticale starch and rice husk reported a pH range of 5.42 to 5.51, classified as slightly acidic. This acidic range is advantageous for the stability and mechanical properties of bioplastics, emphasizing the importance of optimizing the pH of our products to enhance their functional characteristics.

The apparent density analysis revealed that the cocoa shell flour demonstrates significant cohesion and uniformity, with a loose density of 0.35 g/ml and a compacted density of 0.40 g/ml. These values, while slightly lower, are comparable to those reported by Martínez (2023), who found a density of 0.398 g/ml. This suggests that the cocoa shell flour has an average density that falls within the typical range for similar materials.

The rheological properties of the cocoa shell flour revealed a gelatinization temperature of 80 °C and a viscosity of 39 mPa·s. This low viscosity, in comparison to other biopolymers such as cassava and potato starch (Vélez et al., 2021), suggests a reduced resistance to flow. This characteristic may be attributed to the flour's unique composition, which could feature a lower amylose content or a higher proportion of fibrous components. The low viscosity may be advantageous for applications that require greater fluidity, such as formulating food products with a smooth texture or in industrial processes involving pumping and mixing.

Cocoa shell flour, which is rich in native starch, exhibited a water absorption index of 6.75 g/g, slightly higher than the average reported for cocoa shell starch-based bionanocomposites (Noraini et al., 2024). The water solubility of 5.83% and swelling power of 6.9 indicate a moderate interaction with water, which may limit its application in humid environments. These findings align with the general trend observed in starch-based bioplastics, which often exhibit high humidity sensitivity and suboptimal mechanical properties (Oluwasina et al., 2021). However, through chemical modification or the incorporation of reinforcements, it is possible to significantly enhance these properties, thereby broadening the potential applications of this biomaterial.

The moisture content of cocoa shell flour was measured at 24.94%, which is significantly higher than that reported for bioplastic films based on dialdehyde starch (Oluwasina et al., 2021). This elevated moisture level can adversely affect the mechanical properties and stability of the material, promoting microbial growth and degradation. In contrast, the dry matter was calculated at 75.06%, indicating a substantial solid content (Noraini et al., 2024). However, the high ash content of 23.93% suggests a significant concentration of inorganic minerals, which may influence the functional properties of the material. Comparing these findings with those reported by Ramadhan & Handayani (2020) for breadfruit starch, which showed an ash content of only 1.08%, highlights a notable difference that could be attributed to the inherent characteristics of the raw material, as well as the extraction and purification processes employed.

3.2. Bioplastic production process

As shown in Table 1, the bioplastic samples from treatments T1 and T2 exhibited a slightly rough or fibrous texture, characteristic of cocoa shell material. While T1 displayed a more pronounced pigmented brown color, both treatments demonstrated a comparable overall hue. The dried bioplastic samples had a hard and dry texture, reflecting the intrinsic properties of the cocoa shell.

The color of the bioplastic serves as a visual indicator of its cellulose content, with darker shades indicating a higher proportion of cellulose (Azmin et al., 2020). In contrast, bioplastics with lower cellulose concentrations tend to be drier and harder than those with higher cellulose content (Maulida & Maysarah, 2020). Additionally, the presence of cellulose contributes to increased moisture content in the bioplastic, with 100% cellulose bioplastics exhibiting the highest moisture levels (Bhatia et al., 2021). Cellulosic materials naturally absorb and release moisture from their surrounding environment until they reach a state of equilibrium (Wang et al., 2018).

Table 1. Sensory evaluation of the bioplastic film: color, texture, odor and physical properties.

Treatment	Color	Texture	Smell	Physical appearance
T1	Pigmented brown	Dry, hard	Odorless	
T2	Pigmented brown	Dry, hard	Odorless	

Based on the results of the Shapiro-Wilk test, ANOVA was conducted for filtration and solubility, as these variables met the assumption of normality (Table 2). For the remaining variables—moisture, elongation, and strength—which exhibited deviations from normality, the non-parametric Kruskal-Wallis test was employed.

Table 2. Shapiro-Wilk test for assessment of normality.

Variable	n	Mean	SD	W*	p (one tail)
Filtration	6	614.67	255.36	0.86	**0.2535**
Humidity	6	13.33	1.86	0.75	0.0229
Solubility	6	66.11	3.87	0.82	**0.1096**
Elongation	6	8.49	2.64	0.67	0.0020
Endurance	6	27.00	5.21	0.76	0.0251

Although no statistically significant differences in water resistance (measured as filtration) were observed between the bioplastic treatments, T2 demonstrated a slightly higher filtration value of 697.00 mL compared to 532.33 mL for T1, as illustrated in Figure 1. This finding suggests that T2 may possess a marginally more porous or permeable structure, which could influence its barrier properties and potential applications.

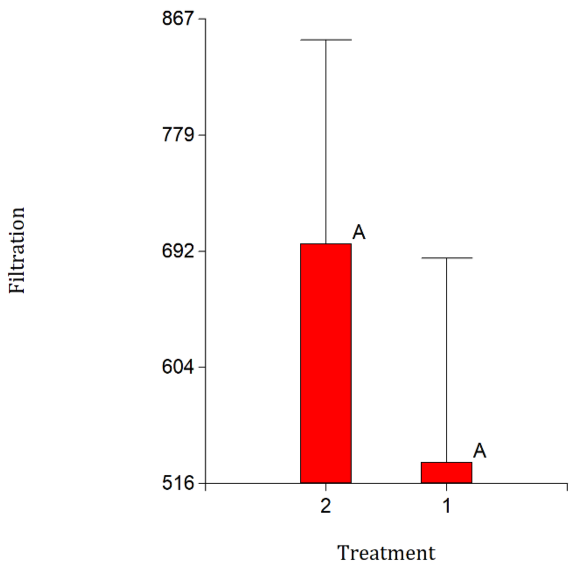

Figure 1. Comparison of filtration values between treatment groups.

While both T1 and T2 displayed reasonable solubility, T2's slight edge might be beneficial in specific applications where solvent compatibility is a critical factor. Further investigation into the underlying reasons for this difference could inform future formulation and optimization efforts.

While no statistical differences were found, T1 consistently outperformed T2 in elongation, strength, and moisture. This suggests a potentially more robust and resilient structure, making T1 better suited for applications demanding higher mechanical performance. Specifically, T1 exhibited an elongation of 10.90%, strength of 31.71%, and higher moisture content of 14.00%, while T2 recorded 6.07%, 22.30%, and 12.67%, respectively (Table 3).

While high moisture content can promote microbial growth and affect the appearance and mechanical properties of the bioplastic (Macêdo et al., 2022), T2 with a moisture content of 12.67% offers a balance between reducing the risk of mold and maintaining microbial activity. This is supported by Azmin et al. (2020), who found a minimum moisture content of 8.01% in cocoa shell bioplastic with 25% cellulose.

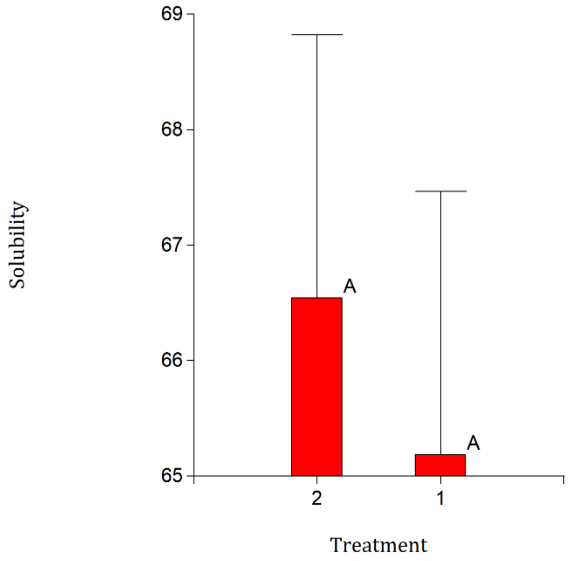

Figure 2. Comparison of solubility values between treatment groups.

Table 3. Kruskal-Wallis test for comparative analysis of elongation, strength and humidity.

Variable	Treatment	N	Means	SD	Medians	H	p
Elongation	1	3	10.90	0.06	10.90	3.86	0.1000
Elongation	2	3	6.07	0.08	6.12		
Endurance	1	3	31.71	1.13	32.12	3.86	0.1000
Endurance	2	3	22.30	0.51	22.56		
Humidity	1	3	14.00	1.73	15.00	0.76	0.7000
Humidity	2	3	12.67	2.08	12.00		

Furthermore, studies have shown a correlation between cellulose concentration and moisture content in bioplastics, with lower cellulose levels resulting in lower moisture uptake (Azmin et al., 2020). In a previous study, Zambrano et al. (2022) produced a bioplastic film from Eichhornia crassipes with a moisture content of 19%. Additionally, increasing the glycerin content of bioplastics has been shown to improve tensile strength. Scanning electron microscopy analysis has revealed that bioplastic films containing 5% glycerin exhibit a more uniform and homogeneous texture (Gallegos-Carrillo et al., 2024).

Elongation, a measure of a bioplastic's flexibility, is crucial for applications requiring flexibility or impact resistance (Budiman et al., 2022). Among the bioplastics tested, T1 demonstrated superior elongation properties, reaching 10.90%. This value significantly exceeded the 3.068% reported by Syuhada et al. (2020) for cassava peel bioplastic and the 6.8% reported by Lestari et al. (2020) for jackfruit seed and rice bioplastics. However, it fell short of the 26.67% achieved by Handayani et al. (2023) using durian seed starch.

The achievement of 31.71% strength by T1 bioplastic represents a significant breakthrough in sustainable materials development. This level of strength indicates the bioplastic's ability to withstand substantial force or stress before breaking or deforming (Oliva & Encinas, 2021). This property is crucial for its potential applications in packaging, automotive parts, and consumer products, demonstrating its potential to enhance durability and performance in these fields (Dawam et al., 2020).

Conversely, T1 exhibited a higher biodegradation rate than T2, with a classification of 77% compared to 64% (Figure 3). These findings suggest that T1 may have a more favorable biodegradation profile, although no statistically significant difference was observed (Table 4). The biodegradability of bioplastics depends on the physicochemical structure of the polymer (Jõgi & Bhat, 2020).

As acetic acid content was the only differentiating factor between treatments, the observed differences in bioplastic properties can be attributed to the influence of acetic acid. While T2 demonstrated superior filtration, solubility, and moisture, T1 exhibited advantages in elongation, strength, and biodegradability. A lower concentration of acetic acid in T1 likely resulted in reduced cross-linking, leading to better flexibility, moisture absorption, and potentially improved biodegradability.

Correlation analysis revealed a moderate positive correlation between filtration and acetic acid ($r = 0.35$, $p < 0.001$), suggesting that both variables increase together. However, a very weak positive correlation was found between acetic acid and solubility (r = 0.21, p = 0.6953), which was not statistically significant. Conversely, a moderate to strong negative correlation was observed between acetic acid and elongation (r = -0.88, p = 0.0213) and strength (r = -0.88, p = 0.0213), indicating that as acetic acid increases, these two variables tend to decrease. While acetic acid and moisture showed a weak negative correlation (r = -0.42, p = 0.4050), it was not statistically significant.

Table 4. Shapiro-Wilk test for the evaluation of the normality of biodegradability.

Variable	n	Mean	SD	W*	P (one tail)
Biodegradability	36	70,00	0.17	0.96	**0.5248**

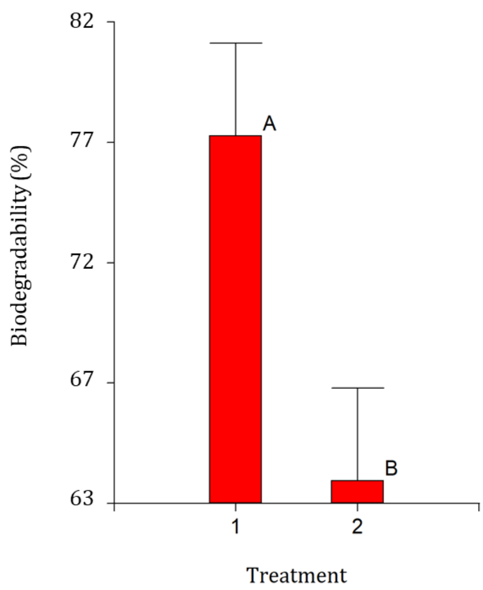

Figure 3. Biodegradation rates of bioplastic samples by treatment.

Acetic acid plays a crucial role in cross-linking the bioplastic structure (Poon et al., 2024). Cross-linking is a chemical reaction that forms bonds between polymer chains, affecting properties such as strength, flexibility, and biodegradability (Bello et al., 2024). The acidic nature of acetic acid could catalyze other reactions between cellulose and other bioplastic components like lignin or hemicellulose, further influencing cross-linking and material properties (Syarif et al., 2022). However, further research is needed to fully elucidate the complex relationship between acetic acid content and bioplastic properties.

Avellán et al. (2020) achieved 89.40% biodegradability after 42 days for corn flour bioplastic. During the bioplastic degradation process, it was observed that a higher glycerin content during bioplastic manufacture correlated with increased degradability. Glycerol's plasticizing effect stems from its ability to reduce intermolecular forces within the flour, thereby decreasing sheet strength. This reduction is due to glycerol's ability to disrupt internal hydrogen bonds (Sernaqué et al., 2020).

4. Conclusions

Bioplastics, made entirely from renewable biological resources, are increasingly recognized as a critical component of a sustainable future. With an annual production of approximately 2 million tonnes, bioplastics offer a promising alternative to conventional, fossil-based plastics (Di Bartolo et al., 2021). These materials contribute to reducing fossil fuel dependency, promoting recycling and biodegradable alternatives, and limiting the use of harmful chemicals in manufacturing processes. Countries such as Japan, Malaysia, Singapore, and South Korea have implemented financial incentives to support the development and adoption of bioplastics (Rosenboom et al., 2022).

Although the bioplastics industry remains small in comparison to conventional plastic production, which reached 360 million tonnes in 2018, it is projected to grow substantially, with an anticipated 40% increase in production over the next five years (Di Bartolo et al., 2021). This growth aligns with the principles of the circular economy, which focuses on minimizing waste and maximizing resource efficiency (Merchan et al., 2022). By using agricultural and food waste as raw materials, the bioplastics industry supports a closed-loop system where materials are continuously recycled and repurposed (Visco et al., 2022). This strategy not only reduces waste but also creates valuable products like bioplastics with characteristics that can further advance sustainable circular bioeconomy goals (Abina et al., 2023; Foschi, et al., 2023).

Our research shows the potential of cocoa shells for bioplastic production. Cocoa shell flour, an agro-industrial by-product, shows significant potential for creating sustainable materials. Despite its high moisture (24.94%) and ash

(23.9275%) content, its rheological and thermal properties, such as low viscosity (39 mPa s) and gelatinization temperature of 80 °C, position it as a promising raw material for various applications. Results from T1 and T2 treatments for obtaining bioplastics demonstrate the flour's versatility. T2 showed a slightly higher filtration value (697.00 mL) compared to T1 (532.33 mL), and for solubility, T2 had a marginal advantage with an average of 66.83% versus T1's 65.38%. While both treatments showed reasonable solubility, T2 might be slightly more suitable for specific solvents or conditions. Conversely, T1 exhibited higher elongation (10.90%), strength (31.71%), and moisture content (14.00%) compared to T2's values of 6.07%, 22.30%, and 12.67%, respectively. Furthermore, T1 had a higher biodegradation rate of 77% versus T2's 64%. Overall, T2 excelled in filtration, solubility, and moisture content, while T1 offered better elongation, strength, and biodegradability.

However, several areas for improvement remain. One key limitation is the extraction and processing of cocoa shell flour, which could be optimized to enhance both the efficiency of the process and the quality of the bioplastic produced. Alternative extraction methods, such as using enzymatic or green solvent techniques, could help improve the yield and properties of the cocoa shell-derived material. Additionally, this study was constrained to only two treatments due to limited financial resources. Future research should expand on this by including more treatments and testing different ratios or combinations of cocoa shell flour with other biodegradable materials, which would provide a broader understanding of how various formulations affect bioplastic performance.

Moreover, future studies could focus on using cocoa shell starch instead of flour as the base material, as starch may offer improved mechanical and thermal properties for bioplastic production, depending on the treatment process. Exploring the feasibility of starch extraction from cocoa shells could open new possibilities for enhancing the material's performance. Additionally, comparative studies with other agricultural waste materials could highlight the relative advantages of cocoa shells in bioplastic applications. Finally, scaling up production and assessing the economic viability of using cocoa shells in large-scale bioplastic manufacturing would be crucial for determining their commercial potential and environmental benefits.

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Funds

This study did not receive external funding.

Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Citation

Burgos Bravo, K.D., Solís Ortiz, S., & Vivas Saltos, H.T. (2024). Cocoa shell bioplastic: a circular path towards sustainability. *Visions for Sustainability*, 23, 10986, 1-20. http://dx.doi.org/10.13135/2384-8677/10986