European Journal of Sustainable Development Research
2025, 9(3), em0309
e-ISSN: 2542-4742
https://www.ejosdr.com/

MODESTUM

Research Article

OPEN ACCESS

Optimizing the production of biodegradable chitosan-glycerol bioplastics from potato starch

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Citation: Nwaka, E., Akomah, U. C., Maduoma, T. U., Ezeokolie, E. D., Menankiti, D. T., Osarenren, E., Donatus, U. D., Orji, C. H., Ekwem, C. H., & Oduaro, I. J. (2025). Optimizing the production of biodegradable chitosan-glycerol bioplastics from potato starch. *European Journal of Sustainable Development Research, 9*(3), em0309. https://doi.org/10.29333/ejosdr/16354

ARTICLE INFO

Received: 28 Jul. 2024

Accepted: 07 Oct. 2024

ABSTRACT

This study explores the optimization of bioplastic production using chitosan and glycerol, employing a central composite design (CCD) to systematically vary the factors of chitosan loading, glycerol loading, and drying time. The primary objective is to enhance the mechanical properties and biodegradability of the resulting bioplastics. The experimental design included 20 runs with varying compositions of chitosan (0.659-2.3409 wt/wt), glycerol (0.159-1.8409 wt/vol), and drying times (4.31821-7.68179 hours). The bioplastics were characterized for tensile strength, elongation at break, and biodegradability after three weeks. The results indicated that the optimal formulation involved a balanced ratio of chitosan and glycerol with a moderate drying time, yielding bioplastics with enhanced tensile strength and biodegradability. Specifically, the optimized sample demonstrated a tensile strength of 2.25 MPa, elongation at break of 22.19%, and biodegradability of 92.31%. These findings were compared with predicted values, showing good agreement and validating the CCD model's efficacy. This study contributes significantly to the field of bioplastic production by providing a detailed analysis of the optimal combination of chitosan and glycerol, highlighting improvements in mechanical properties and biodegradability. It also offers a comprehensive evaluation of actual vs. predicted responses, showcasing the potential of chitosan-glycerol bioplastics as a sustainable alternative to conventional plastics. Future research should focus on scaling up production, exploring additional biopolymer blends, and assessing long-term environmental impacts. This research contributes to and shows the potential of bioplastics to mitigate plastic pollution and advance sustainable materials science.

Keywords: bioplastics, potato starch, glycerol, chitosan, mechanical properties, and biodegradability

INTRODUCTION

Plastics are materials made from organic polymers like nylon, polyethylene, polyvinyl chloride, etc. These polymers are large molecules made from smaller ones called monomers derived from natural materials, such as natural gas, oil and plants. Plastics can either be synthetic, semi-synthetic, or bio-based (Nayanathara Thathsarani Pilapitiya & Ratnayake, 2024). Plastics have found significant usefulness in many industries, especially the packaging industry, where most of the plastics used are synthetic and constitute of over 80% (Ncube et al., 2021). This is because, among other varieties of packaging materials like metals, glass, papers, boards, etc., plastic is discovered to be the best due to its long-life properties such as its ability to be molded, extruded or pressed into solid objects of various shapes. Plus, a wide range of other properties, such as being lightweight, durable, flexible, and inexpensive to produce (Evode et al., 2021; Kibria et al., 2023).

The diversity of polymers and the veracity of their properties are used to make a vast array of products that bring medical and technological advances, energy savings, and numerous other societal benefits (Moshood et al., 2022). As a result, the rate of production of plastics has and is still increasing substantially over the last 60 years, from approximately 0.5 million tons in 1950 to over 400.3 million metric tons in 2022 (Statista, 2023). These plastics eventually

turn into waste and fragment into smaller toxic components that pollute the soil, endanger marine lives, clog up the drains, cause water and sewage to overflow, and even become the breeding grounds for germs and bacteria that spread disease (Amarakoon et al., 2022; Dumbili & Henderson, 2020; Kibria et al., 2023).

Even though several plastic disposal techniques such as landfilling and incineration have emerged, they end up causing more harm than good (Evode et al., 2021; Pandey et al., 2023). For instance, while burning is a good option for plastic waste management, it produces high unhealthy emissions like dioxins and furan that add to environmental problems. When they are used in landfilling, they have the potential to end up in the oceans and water bodies and endanger aquatic species (Kehinde et al., 2020; Kumar et al., 2021). Plastic recycling, however, requires a considerable amount of energy (Sebastian & Paul, 2021). Synthetic plastics are nondegradable and the best way to eliminate them is to not produce them, which is why this study aims to explore the production of biodegradable plastics and statically optimize its production from potato waste by studying the effects of the independent variables (chitosan loading, glycerol and drying time) on the responses (tensile strength, elongation, and biodegradability). Starch from different sources has been studied as a potential film-forming agent, including that from potato, barley, wheat, tapioca, and rice. Thus, it has become a promising alternative material to replace conventional plastics in individual market segments (Hirpara et al., 2021; Makroo et al., 2021).

Biodegradable plastics are plastics that can be decomposed by the action of living organisms, usually microbes, into water, carbon dioxide, and biomass. They are commonly produced with renewable raw materials, microorganisms, petrochemicals, or combinations of all three (Moshood et al., 2022). A unique feature of bio-plastics is that, unlike most conventional plastics, the post-use materials may be treated biologically, e.g., via aerobic composting, to generate carbon and nutrient-rich compost as a soil improver. However, a major challenge in the use and production of biodegradable plastics is that biodegradability is a system property (Atiwesh et al., 2021). This means that whether or not a particular plastic will degrade depends not only on the properties of the plastic but also on the conditions of the immediate environment where it is disposed of; temperature, photodegradation, presence of microorganisms etc. Even so, results indicated that plastics from renewable sources would degrade in a short timeframe of sixty days, whereas those with perishable additives would need a longer degradation time (Ahsan et al., 2023). Despite these benefits, data from the bioplastics market development update 2023 showed that bio-plastic represents approximately 0.5% of the over 400 million tons of plastic produced annually (EUBIO, 2023).

Several studies have investigated the potential of potato starch as a feedstock for bioplastics (Bezirhan & Bilgen, 2019; Eterigho et al., 2017; Hirpara et al., 2021; PoStaTic, 2019). Bezirhan and Bilgen (2019) explored the production and biodegradability of bioplastic from potato peel. Their result showed that the produced potato peel bioplastic had a higher water absorption capacity than commercial bioplastic. The study suggests that these bioplastics from potatoes can be used in the packaging industry. They recommended that the development of mechanical properties should be investigated for the utilization of it in different industrial areas (Bezirhan & Bilgen, 2019). Similarly, Hirpara et al.'s (2021) research on developing biodegradable packaging film from potato starch further explores the optimization of preparation conditions to enhance the film's properties. Their study uses a response surface methodology to analyze the effects of varying starch and glycerol concentrations on the film's physical properties. The results indicate that potato starch-based films exhibit desirable characteristics such as high moisture content and water absorption capacity, making them suitable for specific packaging applications (Hirpara et al., 2021). The PoStaTic (2019) project, supported by the Institut Français des Matériaux Agrosourcés, further explores the structural and morphological optimization of potato starch for bioplastic production. This research aims to identify potato varieties with the most suitable starch properties for bioplastic manufacturing, focusing on in vivo modifications of starch granule size and amylose content. The project's comprehensive approach, utilizing advanced methods such as synchrotron-based X-ray fluorescence microscopy, seeks to improve the plasticization and film-forming properties of potato starch-based bioplastics, potentially leading to significant environmental and economic benefits (PoStaTic, 2019). Eterigho et al. (2017) investigate the synthesis and characterization of potato starch-based plastics using polyvinyl alcohol (PVA) as a cross-linker. Their study finds that increasing PVA concentration enhances the mechanical properties of the bioplastics, such as tensile strength and elongation. The research also demonstrates that these bioplastics are biodegradable, with the PVA/TPS blend degrading in soil, making them a viable alternative to petroleum-based plastics (Eterigho et al., 2017).

While these studies highlight the potential and advancements in bioplastic production from potato starch, there is a need for the optimization of production processes and the enhancement of mechanical and biodegradable properties. This study aims to address these gaps and contribute to to the applied research and development of eco-friendly and economical bioplastics by pursuing the following research objectives:

(1) Produce bioplastics using local potatoes as the main feedstock.

(2) Investigate the solubility and absorption properties of the produced biodegradable plastics in different solvents and visualize the microstructure through Keyence optical digital microscope VH-7000.

(3) perform mechanical tests (tensile stress, strain, elasticity, and Young's modulus) on bioplastic samples produced.

(4) optimize the bioplastic produced using chitosan and glycerol and a central composite design (CCD) to systematically vary the factors of chitosan loading, glycerol loading, and drying time.

METHODOLOGY

The Potato Starch extraction

Potato tubers were sourced from Ihiagwa market, Owerri West, Imo State, while analytical grade glycerol and distilled water were procured from new concept laboratory, Obinze. Approximately 5 kg of potatoes were washed, peeled, and diced before blending with a 1:10 potato-to-water ratio. Ice was added to mitigate the heat generated during the process. The resulting slurry was filtered through a double-layered fabric to extract starch granules. This process was repeated to maximize starch recovery. The extracted starch, along with glycerol (propan-1, 2, 3-triol) and 0.1 mol/dm^3 hydrochloric acid, was employed in subsequent bioplastic production using standard laboratory equipment, including beakers, watch glasses, a heater plate, magnetic stirrer, tripod, gauze, stirring rod, petri dish, indicator paper, eye protection, and cylinders.

Physio-Chemical Characteristics of Potato Starch

The physiochemical properties of the extracted potato starch were determined according to Institute of Professional Analysts of Nigeria standards at New Concept Laboratory, Obinze, Owerri. Parameters assessed included pH, moisture content, ash content, bulk density, gelatinization temperature, and protein content.

pH was measured by dissolving 2 g of starch in 50 ml of distilled water and using a pH meter. Moisture content was determined by drying 5 g of starch in an oven at 100 °C for 4 hours, followed by cooling in a desiccator and calculating the weight loss. The moisture content was calculated using Eq. (1):

$$\%Moisture\ content\ = \left[\frac{(W2 - W3)}{(W2 - W1)}\right] \times\ 100\%, \quad (1)$$

where W1 is the initial weight of the empty crucible, W2 is the weight of the crucible and sample before drying, and W3 is the weight after drying. Bulk density was calculated by measuring the volume occupied by a known weight of starch in a graduated cylinder, as seen in Eq. (2):

$$Bulk\ density, g/ml\ =\ [Weight/(\ volume)]. \quad (2)$$

Gelatinization temperature was ascertained by heating a starch-water suspension while stirring and noting the temperature at which a gel formed. Yield percentage was calculated as the ratio of the weight of extracted starch to the initial weight of potatoes multiplied by 100, as expressed in Eq. (3):

$$\%Yield\ = \frac{Weight\ of\ starch\ obtained\ after\ extraction}{Weight\ of\ potato\ before\ extraction} \times 100\%. \quad (3)$$

Chitosan Production

Periwinkle shells, sourced from Ihiagwa market, Owerri West LGA, Imo State, were washed, dried in a hot air oven at 65 °C for 3 hours, and milled using a domestic blender. The resulting particles were sieved through a 1 mm sieve. Five grammes of the milled shells were mixed with sodium hydroxide (NaOH) in a 1:10 (w/v) ratio, boiled for 10 minutes, and centrifuged at 5,000 g for 10 minutes. The residue was deproteinized with hot water, demineralized with excess 6N HCl, and rinsed with hot water before filtering through the Whatman No. 42 filter paper to obtain crude chitin. Deacetylation involved boiling the crude chitin in a 50% NaOH solution at 100 °C for 4 hours, followed by extensive washing with distilled water and drying to yield chitosan.

Preparation of Biodegradable Plastic Film

To prepare biodegradable plastic film, a constant 5% wt/wt of potato starch was mixed with 100 ml of distilled water and varying amounts of glycerol (0.5%, 1%, and 1.5% wt/vol) using a magnetic stirrer at room temperature for 15 minutes. Each mixture was then combined with different proportions of chitosan powder (1%, 1.5%, and 2% wt/wt) at 400 rpm and heated to 85 °C for 2 minutes until gelatinization occurred. The gelatinized suspension was immediately poured into circular petri dishes and dried in a hot ventilated oven at 60 °C with varying drying times of 4 hours, 4.3 hours, 5 hours, 6 hours, 7 hours, and 7.6 hours according to the experimental design.

Effects of Variables on Prepared Biodegradable Plastic Films

To determine the optimal amount of glycerol used as a plasticizer, its quantity was varied from 0.5 g to 1.8 g, with the starch amount kept constant, and its effect on the tensile strength of the plastic film was measured. Similarly, to find the best amount of chitosan used as a filler, its quantity was varied from 0.65 g to 2.3 g, with a constant starch load, and the impact on tensile strength was evaluated. Additionally, the drying time was varied from 4.3 hours to 7.6 hours to assess its influence on the tensile strength of the prepared biodegradable plastic film.

Properties and Characteristics of Prepared Bioplastic Film and Chemical Properties of Biodegradable Plastic Film

The water absorption resistance of the prepared bioplastic films was evaluated by soaking them in water at room temperature for one hour, drying them with cotton pieces, and weighing them. The percentage of water absorption was calculated using Eq.(4):

$$Water\ absorption\ (\%)\ =\ (Wet\ weight - dry\ weight)/(dry\ weight) \times\ 100. \quad (4)$$

For acid absorption resistance, the bioplastic films were soaked in 1M hydrochloric acid solution, and their weights were recorded at hourly intervals. The percentage of acid absorption was calculated using Eq. (5):

$$Acid\ absorption\ (\%)\ =\ (Wet\ weight - dry\ weight)/(dry\ weight) \times\ 100. \quad (5)$$

Similarly, the base absorption resistance was tested by soaking the films in 1M sodium hydroxide solution and weighing them at regular intervals. The percentage of base absorption was calculated using Eq. (6):

$$Base\ absorption\ (\%)\ =\ (Wet\ weight - dry\ weight)/(dry\ weight) \times\ 100. \quad (6)$$

Moisture absorption resistance was determined by drying the bioplastic films in a desiccator until a constant weight (W1) was achieved, then placing them in a normal atmosphere for 24 hours, and weighing them again (W2). The percentage of moisture absorption was calculated using Eq. (7):

$$Moisture\ absorption\ (\%)\ =\ (W2-W1)/W2\times\ 100, \quad (7)$$

where W1 is the initial weight of the film before exposure and W2 is the weight after exposure.

The solubility test involved placing 0.3 g of bioplastic film pieces in test tubes containing 3 ml of acetone, ethanol, or methanol, and observing solubility at 28-30 °C and 60 °C after one hour. Mechanical properties, including film thickness, tensile strength, and elongation at break, were measured using an Ametek universal tensile testing machine EZ250. Strips of the bioplastic films (5 cm long and 0.2 cm wide) were tested for tensile strength, calculated using Eq. (8):

$$Tensile\ strenght\ Mpa = (Force\ at\ break)/(width \times thickness). \quad (8)$$

The percentage elongation was also calculated, as shown in Eq. (9):

$$\%Elongation\ =\ (L-Lo)/Lo\ \times\ 100, \quad (9)$$

where L is the final length and L0 is the initial length. A CCD model was used to analyze the effects of ingredients on the tensile strength using design-expert 13.

The biodegradability property was tested using the soil burial method. Bioplastic films (3 inches by 3 inches) were weighed (W1), buried in moist soil at a 3-inch depth for one week, and reweighed (W2). The percent weight loss was calculated using Eq. (10):

$$\%Weight\ loss\ =\ (W1-W2)/W1, \quad (10)$$

where W1 is the weight before burial and W2 is the weight after burial.

RESULTS AND DISCUSSION

The aim of this study was to produce biodegradable plastics from potato waste and optimize their production by examining the effects of chitosan loading, glycerol, and drying time on the mechanical and biodegradable properties of the resulting bioplastics. The objectives included investigating the solubility and absorption properties, performing mechanical tests, and comparing bioplastic samples with varying glycerol contents.

The physio-chemical characteristics of potato starch are critical for bioplastic production. The physiochemical properties of the extracted potato starch were determined as a preliminary step in bioplastic production, as shown in **Table 1**. These properties significantly influence the starch's suitability, mechanical strength, and biodegradability as a biopolymer.

The obtained pH of 5.60 indicates a slightly acidic nature of the potato starch, which is within the typical range for starch (Sakkara et al., 2019). The bulk density of 0.62 g/ml suggests a relatively loose packing of starch granules, which can influence the film-forming properties. It is also comparable to the 0.6-0.8 g/mL range reported in similar studies (Bajpai, 2018). The moisture content of 7.50% is essential for starch processing, as it affects gelatinization and film formation. The gelatinization temperature is consistent with the values around 60 °C-74 °C found in literature (Tomasik & Horton, 2012), indicating suitable film-forming properties for bioplastic production. These characteristics suggest that the extracted potato starch is appropriate for bioplastic production, with potential benefits such as improved mechanical strength and biodegradability due to its physio-chemical properties in **Table 1**.

Table 1. Results of physio-chemical characterization of potato starch

S/N	Characteristics	Result
1	Ph	5.60
2	Bulk density (g/ml)	0.62
3	Moisture content (%w/w)	7.50
4	Gelatinization temperature (°C)	62.30

Table 2. Absorption test on plastics films

Samples	Water absorption resistance (%w/v)	Base absorption resistance (%w/v)	Acid absorption resistance (%w/v)	Moisture absorption resistance (%w/v)
1	25.64	Soluble	28.40	32.70
2	30.52	Soluble	18.89	38.66
3	23.84	Soluble	31.65	29.55
4	20.10	Soluble	17.48	23.48
5	28.73	Soluble	24.27	34.72
6	29.00	Soluble	19.90	28.62
7	22.74	Soluble	21.43	25.99
8	23.21	Soluble	26.08	33.04
9	21.17	Soluble	30.00	27.01
10	33.63	Soluble	25.43	26.89
11	27.65	Soluble	23.79	32.03
12	19.86	Soluble	21.34	33.81
13	24.09	Soluble	20.76	28.90
14	27.82	Soluble	26.71	22.25
15	23.77	Soluble	26.96	23.53
16	30.06	Soluble	29.12	32.98
17	28.04	Soluble	19.17	29.77
18	25.00	Soluble	26.75	27.66
19	29.98	Soluble	28.55	24.17
20	20.48	Soluble	24.10	27.58

Effects of Variables on Bioplastic Properties

Absorption test analysis

The absorption properties of the developed bioplastic films in **Table 2**, are key indicators of their suitability for various applications. The study examined the effects of varying glycerol and chitosan content on the absorption properties of bioplastic films.

The water absorption resistance ranged from 19.86% to 33.63%, indicating that the films had varying degrees of hydrophilicity depending on the glycerol and chitosan content. Higher glycerol content generally increased water absorption due to its plasticizing effect, which enhances film

Table 3. Solubility test result on plastics films

Samples	Ethanol 30 °C	Ethanol 60 °C	Methanol 30 °C	Methanol 60 °C	Acetone 30 °C	Acetone 60 °C
1	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
2	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
3	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
4	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
5	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
6	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
7	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
8	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
9	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
10	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
11	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
12	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
13	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
14	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
15	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
16	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
17	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
18	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
19	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble
20	Insoluble	Swell	Insoluble	Insoluble	Insoluble	Insoluble

flexibility but reduces water resistance (Tarique et al., 2021). Chitosan, known for its hydrophilic nature, contributed to this variability as well. The results show that samples 4, 9, and 12 exhibited lower water absorption, suggesting an optimal balance of glycerol and chitosan content that enhances water resistance. Compared to other bioplastics like polylactic acid and polyhydroxyalkanoates, these potato starch-based films show competitive water resistance, making them suitable for applications where moderate water exposure is expected (Naser et al., 2021).

The bioplastic films' resistance to acidic environments showed significant variability, with acid absorption resistance ranging from 17.48% to 31.65%. The resistance to acid absorption is crucial for packaging acidic food products and in biomedical applications where exposure to acidic environments is common. Films with lower glycerol content, such as sample 4 and sample 6, demonstrated better acid resistance, implying that reduced plasticizer content enhances the film's stability in acidic conditions (Hirpara et al., 2021). This makes these bioplastic films suitable for packaging applications that require acid resistance.

Despite their promising properties, the bioplastic films were found to be soluble in alkaline conditions. This solubility is a significant limitation for applications involving exposure to basic substances, such as in agricultural mulches or industrial settings using alkaline solutions. The high solubility indicates that the films rapidly degrade in basic environments, which, while limiting for some applications, could be beneficial for waste management where rapid degradation is desired (Evode et al., 2021).

Moisture absorption resistance varied from 22.25% to 38.66%, influenced by the environmental humidity and film composition. Higher glycerol content generally increased moisture absorption due to its hygroscopic nature, which attracts and retains water molecules (Chen et al., 2022). This characteristic can be a disadvantage in humid environments but could be beneficial for soil moisture retention in agricultural applications. Compared to conventional plastics, which typically exhibit low moisture absorption, these bioplastics offer a balance between biodegradability and functional properties. The optimal formulation for specific applications would need to consider the desired balance between flexibility, strength, and environmental stability.

Solubility test analysis

The solubility test results in **Table 3**, indicate that the films are insoluble in ethanol, methanol, and acetone at 30 °C. However, at 60 °C, the films swell in ethanol but remain insoluble in methanol and acetone. The swelling in ethanol at elevated temperatures suggests partial interaction between the polymer matrix and the solvent, which can be attributed to the plasticizing effect of ethanol (Chen et al., 2014). This characteristic implies that the bioplastic films have good chemical resistance, making them suitable for packaging applications where exposure to solvents at room temperature is common. However, storage and handling conditions should avoid high temperatures in ethanol-rich environments to prevent swelling and potential structural compromise.

Mechanical Properties

Tensile strength

The tensile strength of bioplastic films is a critical measure of their durability and resistance to breaking under tension. From **Table 3**, the tensile strength values range from 1.42 MPa (sample 11) to 2.5 MPa (sample 9), with higher values indicating stronger materials. The ANOVA model analysis revealed that the interaction between chitosan loading (A), glycerol content (B), and drying time (C) significantly influenced tensile strength. Glycerol loading (B) and drying time (C) displayed a significant positive effect, while chitosan loading (A) had a significant negative impact. The interaction graph in **Figure 1**, **Figure 2**, and **Figure 3** displays how the response changes according to each factor from a chosen reference point.

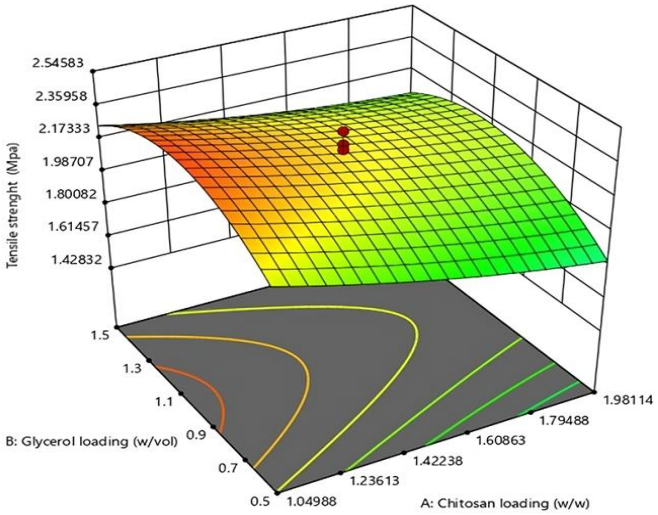

Figure 1. 3D surface plot of tensile strength (Source: Authors' own elaboration)

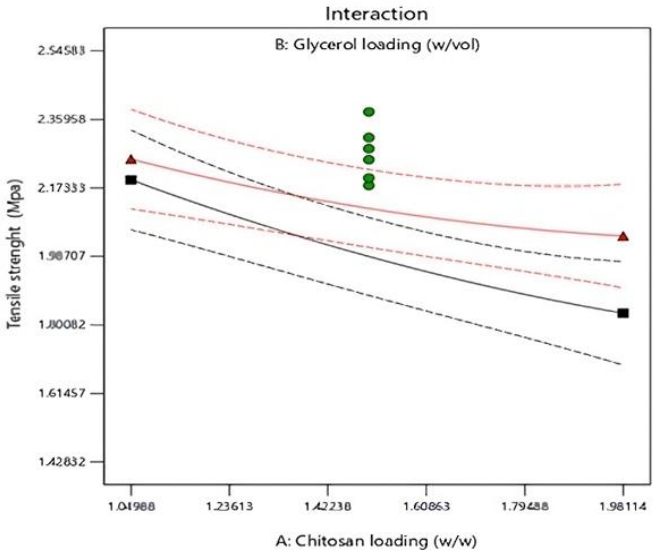

Figure 2. Interaction plot if tensile strength (Source: Authors' own elaboration)

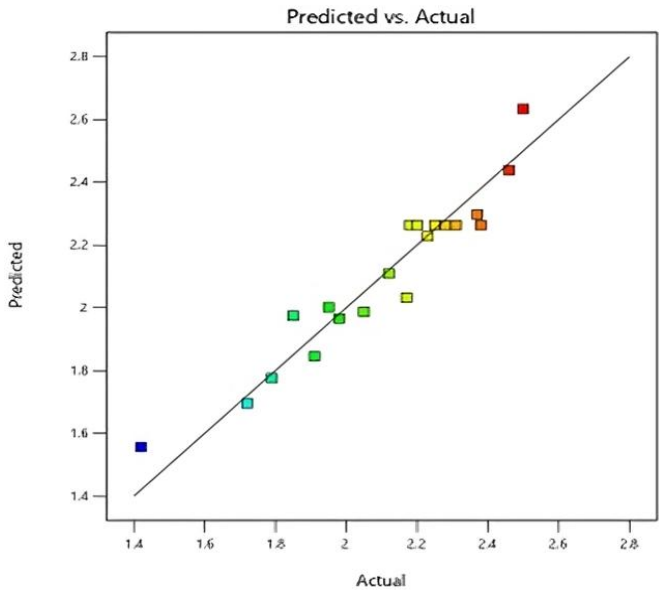

Figure 3. Predicted vs. actual values for tensile strength (Source: Authors' own elaboration)

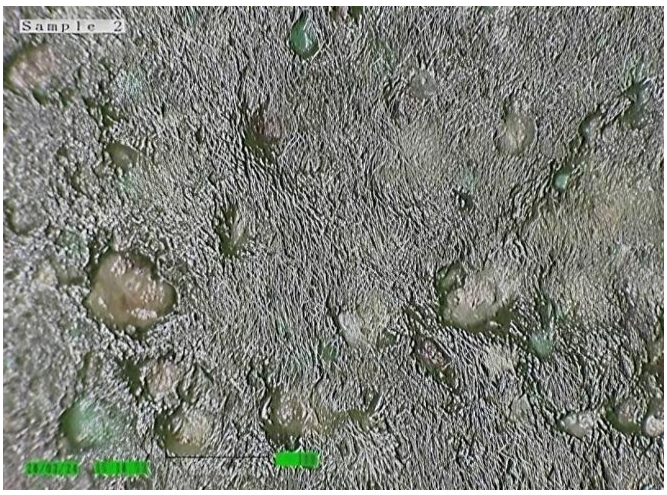

Figure 4. Micrograph of best sample in terms of strength (sample 2) result viewed at X100 resolution (Source: Authors' own elaboration)

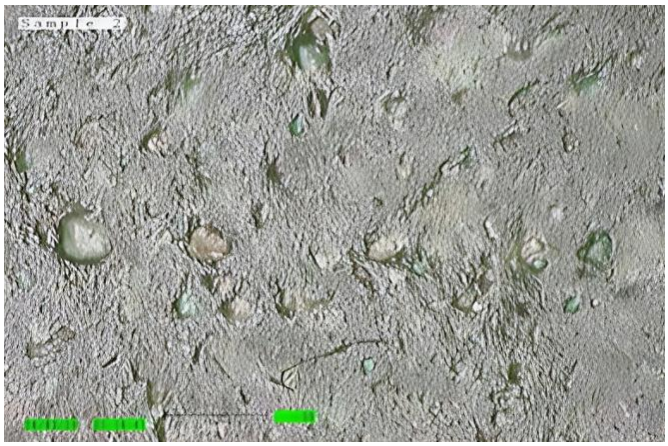

Figure 5. A micrograph of best sample in terms of strength (sample 2) result viewed at X50 resolution (Source: Authors' own elaboration)

In 3D surface plots (**Figure 1**), the response characteristics for this analysis are in the form of maximum response. The correlation between the two variables is that the higher the chitosan loading, the lower the tensile strength. The lower the chitosan loading, the higher the tensile strength. This is due to the function of glycerol as a plasticizer on a calcium based filler. The observed decrease in tensile strength with increasing chitosan content can be attributed to the formation of a more brittle network due to strong interactions between chitosan and starch molecules (Xu et al., 2005).

Samples with higher tensile strength, such as sample 2 (2.5 MPa), are better suited for applications requiring robust and dimensionally stable films. However, these may come at the expense of flexibility but compared to conventional plastics, these bioplastics offer a balance of strength and flexibility, essential for diverse industrial applications (**Figure 4** amd **Figure 5**).

Elongation at break

The elongation at break, indicative of film flexibility, ranged from 13.59% (sample 11) to 24.14% (sample 2) (**Table 4**). According to the ANOVA model, the interaction between

Table 4. Mechanical test results

Samples	Film thickness (mm)	Tensile strength (Mpa)	Elongation at break (%)
1	0.64	2.17	18.00
2	0.50	2.46	24.14
3	0.29	2.05	23.91
4	0.39	1.98	15.20
5	0.71	2.37	18.50
6	0.66	1.72	23.00
7	0.35	1.91	21.76
8	0.28	1.95	20.44
9	0.43	2.50	20.10
10	0.33	2.12	19.95
11	0.29	1.42	13.59
12	0.42	1.79	21.22
13	0.38	1.85	22.00
14	0.47	2.23	23.60
15	0.36	2.31	20.19
16	0.41	2.25	22.19
17	0.28	2.18	22.96
18	0.31	2.28	21.50
19	0.50	2.38	23.79
20	0.25	2.20	21.82

chitosan loading (A) and glycerol content (B) significantly impacted elongation. Similar to the tensile strength analysis, increasing chitosan content resulted in lower elongation at break due to its stiffening effect. Conversely, a positive correlation was observed between glycerol content and elongation, highlighting its role in enhancing film flexibility. This can be explained by the plasticizing effect of glycerol, as it facilitates chain mobility within the polymer matrix, allowing for greater deformation before breaking.

Samples with higher elongation at break values, such as sample 2 (24.14%), are more suitable for applications requiring flexibility, such as wrapping films. It shows high flexibility and suitability for applications requiring deformable materials. But this might be achieved at the cost of lower tensile strength.

The relationship between tensile strength and elongation at break is generally material-dependent. Typically, materials with high elongation exhibit lower tensile strength. The ANOVA model for elongation at break confirmed this trend (22.4247 + 24.2316 × B - 10.335 × AB - 6.04449 × B2).

The positive coefficient for glycerol content (B) indicates its positive influence on elongation, while the negative coefficient for the interaction term (AB) suggests a counteracting effect when combined with chitosan content. This reinforces the need to carefully balance the content of chitosan and glycerol to achieve the optimal combination of strength and flexibility for the desired application (**Figure 6**, **Figure 7**, and **Figure 8**).

Biodegradability

The biodegradability of the bioplastic samples was evaluated based on weight loss over time. All samples exhibited significant degradation, with over 30% weight loss in the first week, 52% by the second week, 80% by the third week, and complete degradation by the fourth week. This rapid biodegradation contrasts sharply with synthetic plastics,

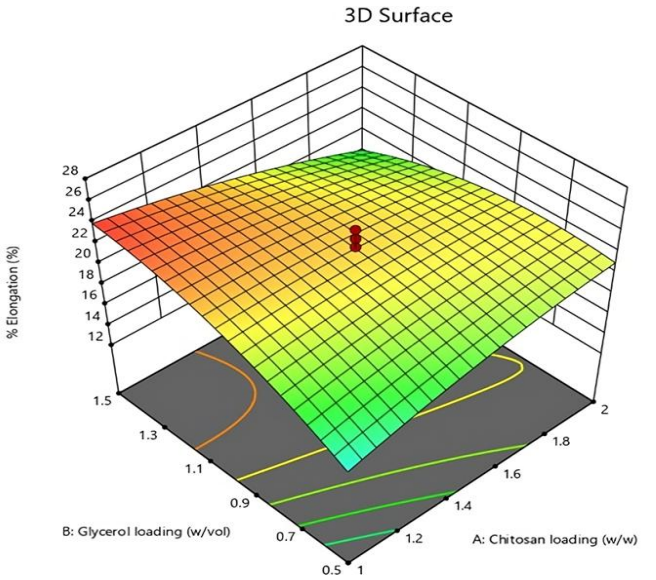

Figure 6. 3D surface plot of elongation break (Source: Authors' own elaboration)

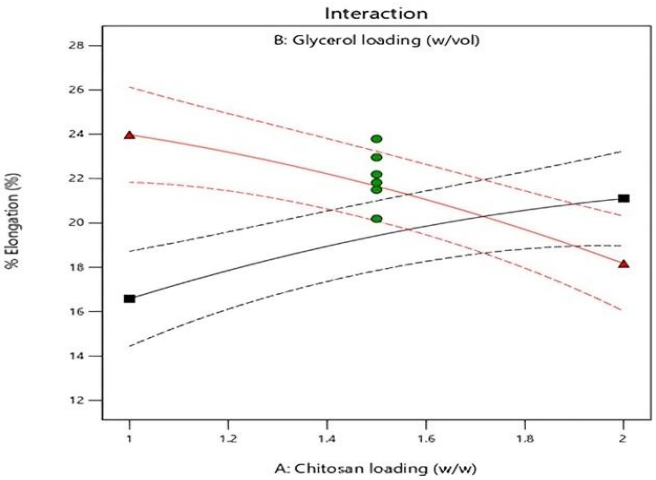

Figure 7. Interaction plot of elongation break (Source: Authors' own elaboration)

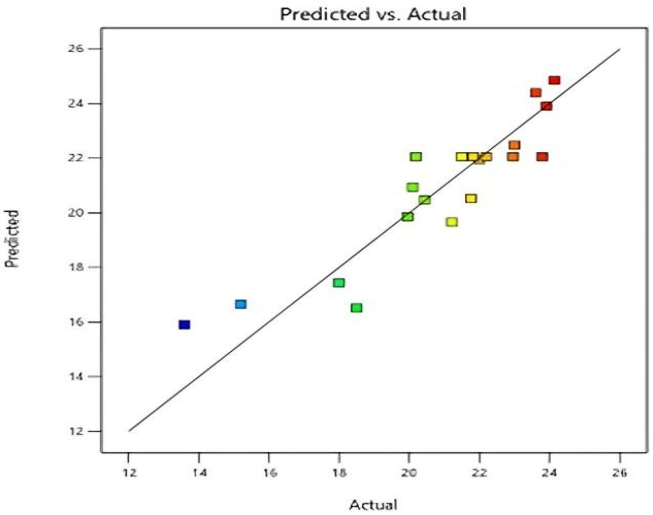

Figure 8. Predicted vs. actual values for elongation break (Source: Authors' own elaboration)

Figure 9. Chart of weight (A) (Source: Authors' own elaboration)

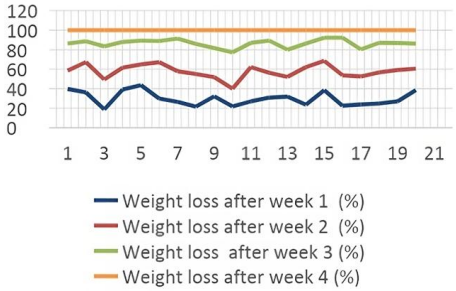

Figure 10. Chart of weight (B) (Source: Authors' own elaboration)

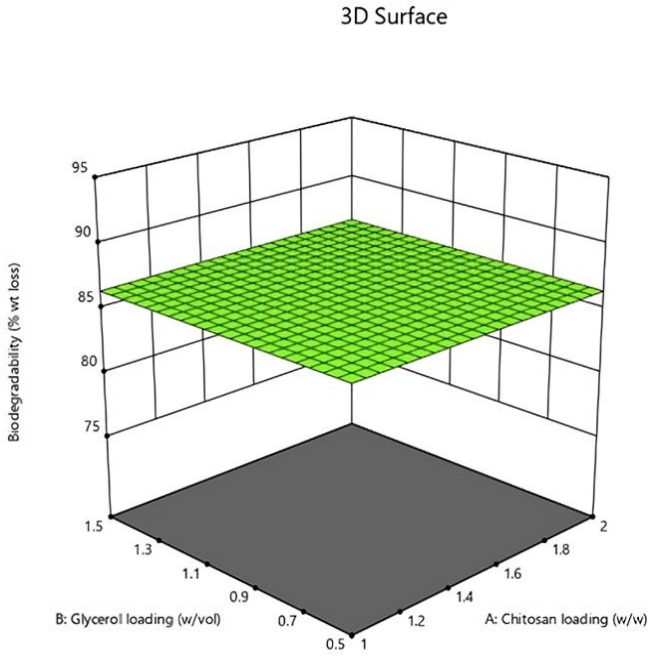

Figure 11. 3D surface plot of biodegradability (Source: Authors' own elaboration)

which can persist in the environment for hundreds of years (**Figure 9** and **Figure 10**).

The ANOVA analysis for biodegradability revealed a lack of significant variation among the samples (F-value = 0.7836, p-value = 0.6720). This suggests that the formulation variables, including chitosan content, glycerol content, and drying time, did not significantly influence the overall biodegradation rate.

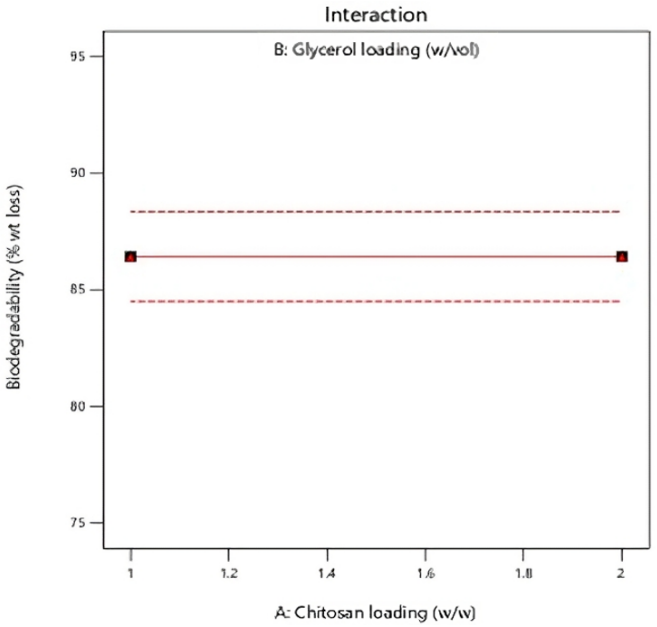

Figure 12. Interaction plot of biodegradability (Source: Authors' own elaboration)

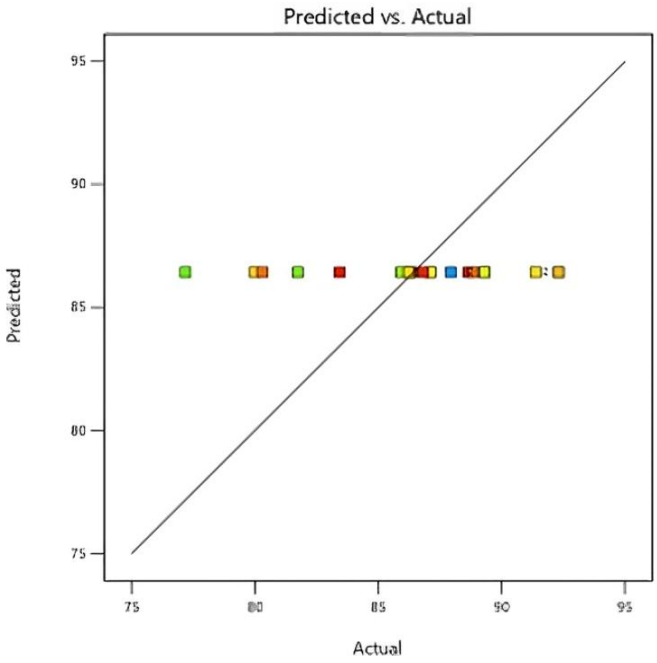

Figure 13. Predicted vs. actual values for biodegradability (Source: Authors' own elaboration)

The interaction plots and 3D surface plots demonstrate that none of the independent variables (chitosan loading, glycerol loading, and drying time) significantly affect the biodegradation rate. This finding indicates that the biodegradability of the films is primarily governed by the inherent properties of potato starch rather than the specific formulation (**Figure 11**, **Figure 12**, **Figure 13**, and **Figure 14**).

Optimization of Bioplastic Production

The optimization of bioplastic production involved analyzing desirable mechanical properties and biodegradability.

Figure 14. Produced biodegradable plastic films (Source: Authors' own elaboration)

Table 5. Composition of various samples

Run (samples)	Factors (variables)		
	Chitosan loading (wt/wt)	Glycerol loading (wt/vol)	Drying time (hour)
1	1	0.5	5
2	1	1.5	7
3	1	1.5	5
4	2	1.5	5
5	1	0.5	7
6	2	0.5	7
7	2	0.5	5
8	2	1.5	7
9	0.659104	1	6
10	2.340900	1	6
11	1.5	0.159104	6
12	1.5	1.840900	6
13	1.5	1	4.318210
14	1.5	1	7.681790
15	1.5	1	6
16	1.5	1	6
17	1.5	1	6
18	1.5	1	6
19	1.5	1	6
20	1.5	1	6

Composition of various samples

The composition of the bioplastic samples was varied based on three factors: chitosan loading (wt/wt), glycerol loading (wt/vol), and drying time (hours). **Table 5** provides a detailed breakdown of these compositions:

Evaluation of predicted vs. actual responses

The results of predicted vs. actual responses are summarized in **Table 6**. This comparison helps validate the CCD model and assess its predictive accuracy for tensile strength, elongation at break, and biodegradability.

The result showed that the optimal combination of glycerol, chitosan, and drying time based on the evaluation of predicted vs. actual responses, includes:

- **Chitosan loading:** 1.5 wt/wt
- **Glycerol loading:** 1 wt/vol
- **Drying time:** 6 hours

This formulation achieved a balance between tensile strength, elongation at break, and biodegradability. The optimized bioplastic films exhibit tensile strengths ranging from 1.72 to 2.5 MPa, elongation at break values from 13.59% to 24.14%, and complete biodegradation within four weeks.

Table 6. Predicted vs. actual response of tensile strength, elongation at break, and biodegradability

Samples	Actual responses			Predicted responses		
	Tensile strength (Mpa)	Elongation at break (%)	Biodegradability after 3 weeks (%wt lose)	Tensile strength (Mpa)	Elongation at break (%)	Biodegradability after 3 weeks (%wt lose)
1	2.17	18	86.29	2.03	17.44	86.43
2	2.46	24.14	88.65	2.44	24.85	86.43
3	2.05	23.91	83.44	1.99	23.91	86.43
4	1.98	15.20	87.95	1.97	16.66	86.43
5	2.37	18.50	89.30	2.30	16.51	86.43
6	1.72	23.00	88.85	1.70	22.48	86.43
7	1.91	21.76	91.38	1.85	20.52	86.43
8	1.95	20.44	85.93	2.00	20.48	86.43
9	2.50	20.10	81.74	2.63	20.94	86.43
10	2.12	19.95	77.19	2.11	19.86	86.43
11	1.42	13.59	87.09	1.56	15.90	86.43
12	1.79	21.22	89.30	1.78	19.66	86.43
13	1.85	22.00	80.00	1.97	21.96	86.43
14	2.23	23.60	86.34	2.23	24.39	86.43
15	2.31	20.19	92.31	2.26	22.05	86.43
16	2.25	22.19	92.31	2.26	22.05	86.43
17	2.18	22.96	80.32	2.26	22.05	86.43
18	2.28	21.50	87.12	2.26	22.05	86.43
19	2.38	23.79	86.81	2.26	22.05	86.43
20	2.20	21.82	86.23	2.26	22.05	86.43

The CCD model provided a robust framework for optimizing the bioplastic formulation. The model accurately predicted the mechanical properties and biodegradability, with the predicted values closely matching the actual responses. The optimal formulation's tensile strength and elongation at break suggest that the bioplastic is both strong and flexible, making it suitable for various applications, including packaging and agricultural films. The rapid biodegradation rate is a significant advantage over synthetic plastics, which are notorious for their persistence in the environment and contribution to pollution (Atiwesh et al., 2021).

The influence of chitosan and glycerol on the mechanical properties and biodegradability highlights the importance of selecting appropriate biopolymer and plasticizer concentrations. Higher glycerol content generally improves flexibility but may reduce tensile strength, while chitosan enhances mechanical strength but may decrease elongation at break.

CONCLUSION

This study successfully developed biodegradable plastic films using locally sourced potato starch as a renewable feedstock. The study explored the optimization of film properties through a CCD experimental approach and represents a significant advancement in the field of bioplastics that offers a potential alternative to environmentally detrimental synthetic polymers. The results demonstrated the feasibility of producing bioplastic films with acceptable mechanical properties, including tensile strength and elongation, while achieving rapid biodegradation. The films exhibited desirable properties such as good solubility resistance to common organic solvents, indicating their potential for various applications. Also, these findings are consistent with previous studies (Bezirhan & Bilgen, 2019; Eterigho et al., 2017; Hirpara et al., 2021; PoStaTic, 2019), but extends upon previous research by comprehensively investigating the influence of multiple variables on film properties and employing statistical optimization techniques to identify optimal formulations, which indicate that the optimal combination of 1.5 wt/wt chitosan, 1 wt/vol glycerol, and a drying time of 6 hours yields bioplastics with superior tensile strength and elongation at break while maintaining excellent biodegradability. Compared to similar studies, such as those focusing on potato starch-based bioplastics, our findings demonstrate a more balanced improvement in both mechanical properties and environmental performance (Bezirhan & Bilgen, 2019; Eterigho et al., 2017; Hirpara et al., 2021; PoStaTic, 2019). However, the results showed that the optimized conditions were at the boundaries of the CCD. This phenomenon often indicates that the response surface is not well-defined within the interior of the design space, suggesting that the optimal conditions for the desired properties (in this case, mechanical strength and biodegradability) may be influenced more by extreme values of the formulation variables rather than a balanced combination of them. In our case, the boundary conditions may reflect the inherent properties of the materials used (chitosan, glycerol, and potato starch) and their interactions. For instance, higher chitosan content could enhance the mechanical strength of the bioplastics, while varying glycerol levels might affect flexibility and biodegradability. The lack of significant variation among samples, as indicated by the ANOVA analysis (F-value = 320, p-value = 0.6720), implies that while the formulation variables were systematically varied, their impact on biodegradation was minimal, and the inherent properties of potato starch played a more crucial role. This finding emphasizes the importance of understanding the material properties and their interactions when designing bioplastics. It also suggests that further exploration of the formulation space, possibly beyond the current boundaries, could yield additional insights into optimizing bioplastic properties. Future studies might consider varying other factors or using different materials to explore their effects on the performance of biodegradable films.

Author contributions: EN: conceptualization, software, formal analysis, investigation, resources, writing – original draft, writing – review & editing; **UCA:** conceptualization, software, formal analysis, investigation, resources, writing – original draft, visualization; **TUM:** methodology, software, formal analysis, investigation, writing – original draft, writing – review & editing; **EDE:** methodology, investigation, resources, writing – review & editing, supervision; **DTM & EO:** data curation; Uchechukwu Divine Donatus: validation, visualization, supervision; **CHO:** software, project administration; **CHE:** investigation, resources, **IJO:** validation, supervision, project administration. All co-authors agree with the results and conclusions.

Funding: No funding source is reported for this study.

Ethical statement: The authors stated that the research did not involve human or animal subjects, nor did it require any procedures that would necessitate ethical approval. The authors further stated that the study focused solely on the formulation and characterization of bioplastic materials, utilizing standard laboratory techniques and materials that are not subject to ethical review. As such, the authors confirm that no ethical approval was required for this work.

Declaration of interest: No conflict of interest is declared by the authors.

Data sharing statement: Data supporting the findings and conclusions are available upon request from corresponding author.

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