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BIOLOGY

Green plantain (*Musa paradisiaca* L.) starch modified by natural fermentation and calendula (*Calendula officinalis* L.) essential oil for biodegradable and sustainable bioplastics

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Amido de banana-da-terra verde (*Musa paradisiaca* L.) modificado por fermentação natural e óleo essencial de calêndula (*Calendula officinalis* L.) para obtenção de bioplásticos biodegradáveis e sustentáveis

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Green plantain (*Musa paradisiaca* L.) starch modified by natural fermentation and calendula (*Calendula officinalis* L.) essential oil for biodegradable and sustainable bioplastics

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ABSTRACT: This study developed biodegradable films from native green plantain (*Musa paradisiaca* L.) starch, modified by natural fermentation, with different concentrations (0.2, 0.4, and 0.6%) of an emulsion of calendula (*Calendula officinalis* L.) extract. The starch modification and the corporation of the emulsion were evaluated with respect to the films' properties and biodegradability. Significant changes in pH, acidity, and amylose (37.52% to 31.33%) during fermentation were attributed to organic acid production. The addition of extract emulsion resulted in a decrease in water vapor permeability (89%) and significant differences in solubility (32.66%-16.47%). The mechanical analysis indicated a lower modulus of elasticity (39.30 MPa) at higher emulsion concentration, which influenced the maximum stress and deformation of the films. Scanning electron microscopy (SEM) showed coalescences in films with higher emulsion concentrations. The antioxidant capacity measured by the FRAP method showed a significant difference compared to the control (1.09%-1.94%), but the modified starches did not differ from each other. The addition of emulsion improved biodegradability, with treatment F0.6 showing the highest degradation. These results indicated that fermentation-induced starch modification, combined with the incorporation of calendula extract, influences the physicochemical, mechanical, and biodegradation properties of the films. Changes in barrier performance and biodegradability suggested potential applicability as sustainable materials for reducing plastic waste, particularly in applications requiring controlled degradation and functional activity.
Key words: biodegradability, biopolymer, emulsion.

Amido de banana-da-terra verde (*Musa paradisiaca* L.) modificado por fermentação natural e óleo essencial de calêndula (*Calendula officinalis* L.) para obtenção de bioplásticos biodegradáveis e sustentáveis

RESUMO: Este estudo teve como objetivo desenvolver e caracterizar filmes biodegradáveis à base de amido de banana-da-terra verde (*Musa paradisiaca* L.) modificado por fermentação natural e incorporados com diferentes concentrações de emulsão de extrato de calêndula (*Calendula officinalis* L.), avaliando suas propriedades físico-químicas, mecânicas, estruturais, antioxidantes e biodegradabilidade. Para realizar o objetivo, foi desenvolvido filmes biodegradáveis a partir de amido verde nativo de banana-da-terra (*Musa paradisiaca* L.) modificado por fermentação natural, com diferentes concentrações (0,2, 0,4 e 0,6%) de emulsão de extrato de calêndula (*Calendula officinalis* L.). Além disso, a modificação do amido e a incorporação da emulsão foram avaliadas quanto às propriedades e à biodegradabilidade dos filmes. Os resultados mostram que as mudanças significativas no pH, na acidez e na amilose (de 37,52% para 31,33%) durante a fermentação foram atribuídas à produção de ácido orgânico. A adição de emulsão de extrato resultou na diminuição da permeabilidade ao vapor de água (89%) e em diferenças significativas na solubilidade (32,66%-16,47%). Do mesmo modo, a análise mecânica indicou um módulo de elasticidade menor (39,30 MPa) à medida que aumentava da concentração de emulsão, o que influenciou a tensão máxima e a deformação dos filmes. A microscopia eletrônica de varredura (SEM) revelou coalescências nos filmes com concentrações mais elevadas de emulsão. A atividade antioxidante, medida pelo método FRAP, apresentou diferença significativa em comparação ao controle (1,09%-1,94%), mas os amidos modificados não diferiram entre si. Ademais, a adição de emulsão melhorou a biodegradabilidade, com o tratamento F0.6 apresentando a maior degradação. Esses resultados indicam que a modificação do amido induzida pela fermentação, combinada com a incorporação de extrato de calêndula, influencia as propriedades físico-químicas, mecânicas e de biodegradação dos filmes. Portanto, as alterações no desempenho de barreira e na biodegradabilidade sugerem potencial de aplicação como materiais sustentáveis para a redução de resíduos plásticos, particularmente em aplicações que requerem degradação controlada e atividade funcional.
Palavras-chave: biodegradabilidade, biopolímero, emulsão.

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INTRODUCTION

Plastic packaging is essential for food preservation and availability, ensuring safety and accessibility through its practicality, low cost, and durability. However, in recent decades, production and consumption of this packaging have increased, with production exceeding 350 million tons in 2018 (MOHERY et al., 2024; SHAIKH et al., 2021). Consequently, this production contributed to environmental pollution, since most plastics are disposed of improperly and only a fraction is recycled. In addition, these conventional packaging materials are derived from petroleum, a finite resource, making high demand unsustainable due to the limited supply of raw materials (KUMAR et al., 2021; WANG et al., 2024).

Therefore, sustainable options, such as biopolymers derived from renewable sources, arise as promising alternatives. Among the options, starch stands out for its versatility and has been widely studied in the production of biodegradable films capable of mitigating the impacts of synthetic plastics (BANGAR et al., 2022; OJOGBO et al., 2020; WANG et al., 2024). The use of starches, especially those derived from unconventional sources, such as green plantain starch, represents a significant advance in the packaging industry.

Green plantain represents a promising non-conventional source for starch extraction, contributing to the mitigation of post-harvest losses by valorizing fruits that do not meet commercial quality standards. A considerable proportion of bananas produced globally is discarded due to defects in size, appearance, or overripening, reinforcing the importance of alternative uses such as starch extraction and flour production (VIANA et al., 2022). Such fruits can be effectively redirected to industrial processing due to their high starch content, which can exceed 70% on a dry basis, with relatively elevated amylose levels (around 20-35%), depending on cultivar and agronomic conditions. This relatively high amylose fraction, compared to conventional starch sources, confers favorable functional and physicochemical properties, including suitable gelatinization behavior, high paste viscosity, and the ability to form cohesive and continuous matrices. These characteristics are related to its semi-crystalline structure and amylose-amylopectin organization, which directly influence film formation, retrogradation, and barrier performance (BELLO-PÉREZ et al., 1999; CHÁVEZ-SALAZAR et al., 2017; PELISSARI et al., 2019). In particular, the amylose content contributes to the formation of films with improved mechanical strength, flexibility, and structural integrity, as well as enhanced resistance to solubilization (SARTORI & MENEGALLI, 2016). Furthermore, unripe plantain starch is recognized for its high resistant starch content (47-57%), which can influence both functional and nutritional properties (MORENO-ZARAGOZA et al., 2025). These attributes make plantain starch an attractive candidate for the development of biodegradable packaging materials and related applications.

However, native starch has limited mechanical properties, which require modification. Among the modifications that can be made, natural fermentation stands out as a viable alternative, promoting structural changes, such as changes in the amylose-amylopectin ratio, causing the modification of starch granules by enzymes and organic acids, affecting the gelatinization, paste, and texture properties that improve its functional characteristics (SIDDIQUI et al., 2023).

In addition to modification, the incorporation of essential oils into biodegradable film formulations has appeared as a promising alternative for imparting bioactive properties, such as antimicrobial and antioxidant characteristics, extending the shelf life of foods, and reducing the use of chemical additives (BHANDARI et al., 2022). Among the essential oils investigated, calendula essential oil (*Calendula officinalis*) stands out as a widely used plant-derived product in the cosmetic and pharmaceutical industries due to its broad availability and low cultivation cost. The essential oil is rich in bioactive compounds, including flavonoids, carotenoids, terpenoids, and polyphenols, which confer antioxidant and antimicrobial properties. These characteristics make it a promising candidate for introducing bioactive functionality into starch-based films; however, reports on its application in biodegradable starch-based films remain limited (AK et al., 2021; WAWOCZNY et al., 2025).

This application is performed by emulsification, which allows dispersion of these hydrophobic compounds, thereby favoring their use in active packaging (DAMMAK et al., 2019). In this context, modifications via natural fermentation and the incorporation of essential oil emulsions represent viable alternatives to reduce dependence on non-renewable resources while imparting bioactive properties to the films, thereby enhancing their potential as more sustainable materials.

Considering the potential of green plantain starch for film production and natural fermentation as a modification strategy to address key limitations of polysaccharide-based films, such as high solubility

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and limited mechanical and thermal resistance, the incorporation of calendula essential oil, known for its bioactive properties, enhances the functionality of these materials. The combination of fermentation and essential oil emulsions thus provides a means to tailor film properties and introduce bioactive features, contributing to the development of more sustainable alternatives to conventional materials. Accordingly, this study produced biodegradable films based on plantain starch (*Musa paradisiaca* L.) modified by natural fermentation and incorporating a calendula extract emulsion (*Calendula officinalis* L.) to evaluate the effects of these strategies on film properties.

MATERIALS AND METHODS

Materials and reagents

Plantain (*Musa paradisiaca* L.) fruits were purchased in the southwest region of Bahia, Brazil. Glycerol and Tween 80 (≥ 90% purity; LabSynth, Diadema, SP, Brazil) and calendula extract (*Calendula officinalis* L.) (Terra Aromas e Sabores, Nova Lima, MG, Brazil) were also used.

Extraction of green plantain starch

Starch was extracted according to the methodology described by MOORTHY (1991), with some modifications. The green plantain was SANITIZED in a sodium hypochlorite solution (100 ppm) for 15 min, peeled, and cut into 3 mm slices. Subsequently, they were weighed and crushed in a blender with the addition of (0.03 mol L^{-1}) ammonium hydroxide, solution in a 1:7 (m/v) ratio to avoid enzymatic browning and facilitate the release of starch. The crushed mass was filtered through an organza fabric and sieved through 20-mesh metal sieves. The filtrate was then left to rest, allowing the starch to decant. The supernatant was discarded, and the starch was suspended in water. This process was repeated until a clean, transparent supernatant was obtained, with completely white starch at the bottom of the container. The product obtained was oven-dried for 24 hours at 40 ºC. The extracted starch was weighed to calculate the yield, sieved to make the granules uniform, and stored in airtight packaging to prevent moisture absorption. The mass of the fruit pulp and the mass of the starch obtained after extraction and drying were used to calculate the yield of green plantain starch.

Modification by natural fermentation

The starch was subjected to natural fermentation under conditions defined through preliminary assays. Initially, a screening test was conducted at 25 ºC for 30 days, during which the pH and titratable acidity of the fermentation supernatant were monitored every five days. At the end of this period, no significant differences ($P > 0.05$) were observed during fermentation times according to Tukey's test, indicating limited progression of the fermentation process under these conditions. A second preliminary assay was performed at 40 ºC for 35 days under the same monitoring conditions. In this case, a reduction in pH and an increase in titratable acidity were observed only at the end of the fermentation period (35^{th} day), along with the development of a characteristic fermented starch odor, indicating that fermentation had occurred and confirming microbial activity and metabolite production. Based on these results, the fermentation process was conducted at 40 ºC for 35 days.

A starch-water suspension was prepared at a 1:2 (w/v) ratio by dispersing 250 g of starch in 500 mL of distilled water. The mixture was placed in a closed plastic system and maintained under controlled temperature conditions in a B.O.D. incubator (LOGEN, model LG340FT220-RBC). To promote homogenization and controlled oxygen availability, the system was manually agitated every two days. This periodic agitation was performed to avoid sedimentation and to maintain partially aerobic conditions, which can influence microbial activity and the production of organic acids during fermentation. After 35 days, the fermented starch was separated from the supernatant and dried in a forced-air oven at 40 ºC for 24 h.

Characterization of native and modified starch

Moisture, ash, and protein contents were determined according to AOAC methods 925.10, 923.03, and 991.20, respectively (AOAC International, 2016), while lipid content was quantified following BLIGH & DYER (1959). The apparent amylose content was determined by the colorimetric iodine-binding method described by MARTÍNEZ & CUEVAS (1989). Additionally, starch morphology was evaluated using a scanning electron microscope (SEM; JSM-6610, JEOL, Tokyo, Japan) operated at 10 kV, with samples mounted on conductive double-sided copper tape and sputter-coated with gold using a Denton Vacuum Desk V system (Moorestown, NJ, USA).

Preparation of calendula extract emulsion

The emulsion was prepared using the ultrasonic emulsification technique, according to the methodology by PRAKASH et al. (2020), with

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canola oil, calendula extract, and Tween 80 added at a 5:1:6 (v/v/v) ratio to 88 mL of distilled water. The emulsion was analyzed under an optical microscope to verify the formation of the encapsulation.

Film production

The films were prepared by casting, following the methodology of FAKHOURI et al. (2007) with modifications. The film-forming solutions were prepared with native and modified green plantain starch (3% w/v, 8.38 g), glycerol (40% w/w, 3.35 g, relative to starch mass), distilled water (250 mL), and calendula extract (0.2, 0.4, and 0.6% w/w, relative to starch mass). The suspension (starch and distilled water) was homogenized with gentle stirring on a magnetic stirrer for 30 min to achieve complete starch hydration. Then, the glycerol plasticizer was added and heated to 80 ºC under constant manual stirring with a glass rod until starch gelatinization was achieved (20 min). Subsequently, the film-forming solution was sonicated in a digital ultrasonic bath (SoniClean Sanders Medical, Santa Rita do Sapucaí, MG, Brazil) to remove any bubbles. The emulsion was incorporated after the film-forming solution was sonicated and then subjected to a second sonication, so that the extract emulsion incorporated the polymeric matrix of the film. The film-forming solution was then poured onto a rectangular glass plate and dried at 35 ºC in an air-circulation oven for 24 h to remove the solvent. After drying, the films were conditioned in an environment with a 50% (±5%) relative humidity using a desiccator containing a saturated solution of sodium bromide (NaBr) for 48 h before analyses. The films were produced in three replications, totaling 15 samples.

Film characterization

Thickness

Film thickness was determined using a digital micrometer (Mitutoyo, Suzano, SP, Brazil) with an accuracy of 0.001 mm. The measurements were determined as the average of 10 random locations on the films.

Solubility in water

Film solubility was determined in triplicate according to the method described by GONTARD et al. (1994), with adaptations. The initial mass of the film sample (2 cm x 2 cm) was determined after drying in an oven at 105 ºC for 24 h. The dried films were immersed in 50 mL of distilled water in an Erlenmeyer flask and stirred slowly in a metabolic bath (Dubnoof SL-157/30E, Piracicaba, SP, Brazil) for 24 h at 24 ºC. The samples were then removed from the Erlenmeyer flasks and dried under the same conditions described previously to determine the final mass. Solubility was expressed as a percentage of solubilized material.

Water activity

Water activity (a_w) was measured using a water activity analyzer (Aqualab, 3TE, Washington, USA). The samples were placed in an appropriate container and then placed in the equipment for reading at 25 ºC.

Water vapor permeability (WVP)

Film water vapor permeability was measured using the capsule method described in ASTM 96 (2016), with modifications. The films were cut into 6.5 cm discs, sealed in permeation capsules containing 18 g of silica, and placed in a desiccator containing water at 24 ºC and 100% relative humidity. The mass gain was determined by weighing the permeation capsules on an analytical balance at 1 h intervals for 10 h. The results allowed the construction of plots of test time versus weight gain. The linear segment of the curve corresponds to steady-state water-vapor transfer through the material. The slope coefficient is determined from the linearity of the curve (mass/time). The slope coefficient divided by the specimen's exposed area is the permeability rate (WVPR).

Permeability was determined using the WVPR value, as described in equation 1.

$$WVP = \frac{WVPR\ x\ e}{Psat\ x\ (RH_{1-}\ RH_2)} \quad (1)$$

Where: e is the thickness of the bioplastic (mm), $Psat$ is the saturation pressure at the test temperature (3.169 kPa), RH_1 is the relative humidity of the desiccator (100%), and RH_2 is the relative humidity inside the capsule (0%).

Mechanical properties

Film tension tests were conducted on a texture analyzer (CT3, Brookfield, USA) with a 25 kg load cell coupled with a TA-DAGA Dual Grip Assembly claw, according to the methodology described by ASTM (2010). Samples measuring 25 mm x 100 mm were used for the tensile test.

Structural morphology of films by scanning electron microscopy – SEM

Film micrographs were obtained under the same conditions described for starches at LabMic-UFG using a scanning electron microscope (SEM)

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(JSM-6610, JEOL, Tokyo, Japan) equipped with EDS and Thermo Scientific NSS Spectral Imaging at 10 kV. The films were previously cut with liquid nitrogen and placed on a conductive double-sided copper tape, which was fixed to a sample holder, to generate images from transverse and superficial perspectives. Subsequently, a conductive gold coating was applied using the gold film deposition system Desk V, manufactured by Denton Vacuum LLC (Moorestown, New Jersey, USA).

Analysis of films by Fourier-transform infrared spectroscopy - FTIR

The analysis of the functional groups in samples of native starch films and those modified by natural plantain fermentation was carried out by a Fourier-transform infrared spectrophotometer (FTIR-ATR) (Cary 630 Agilent Technology Inc.) equipped with attenuated total reflectance (ATR) with diamond crystal cell and deuterated triglycine sulfate (DTGS) detector. The spectra were obtained in the mid-infrared region, 4,000 to 600 cm^{-1}, at absorbance with a 4 cm^{-1} resolution and 64 scans.

Optical properties

Film color analysis was measured using a portable colorimeter (CR400, Konica Minolta, Osaka, Japan). The CIELAB system was used to measure lightness (L^* 0: dark, 100: white), a^* (negative value: green, positive value: red), and b^* (negative value: blue, positive value: yellow). The L^*, a^*, and b^* values were obtained with three readings in equidistant regions.

Determination of antioxidant activity

Total antioxidant capacity by free radical capture – DPPH

The DPPH (2,2-diphenyl-1-picrylhydrazyl) test was used to evaluate the in vitro antioxidant capacity of the films. Films prepared with native and modified starch and at different emulsion concentrations (0.2, 0.4, and 0.6%) were cut into small pieces and mixed with 3 mL of 80% methanol. The mixture was vortexed for 3 min and left at room temperature for 3h. Then, it stirred again for 3 min. The obtained supernatant was analyzed for antioxidant capacity by measuring its ability to scavenge the DPPH radical. An aliquot of the supernatant (0.1 mL) was mixed with 3.9 mL of 0.06 mM DPPH in 80% methanol. The mixture was vigorously stirred in a tube shaker for 1 min and then left to stand at room temperature in the dark for 50 min. The absorbance was measured at 515 nm using a digital spectrophotometer (SHIMADZU UV-1700, São José dos Pinhais, PR, Brazil).

Determination of antioxidant capacity by the ferric reducing method - FRAP

The ferric ion-reducing antioxidant power methodology was performed as described by SANTOS et al. (2024). The ferric ion-reducing antioxidant power (FRAP) assay is based on the production of the Fe^{2+} ion from the reduction of the Fe^{3+} ion present in the 2,4,6-tripyridyl-s-triazine (TPTZ) complex. A standard curve was constructed with ferrous sulfate solutions at concentrations of 500 to 1500 µM. The calculation was based on the straight-line equation obtained by adjusting the standard curve, and the results were expressed as mg Fe^{2+}/g of extract.

Biodegradability analysis of films

The biodegradability analysis of the produced films was performed according to the methodology described by MARTUCCI & RUSECKAITE (2009), with adaptations. The films were cut into 2 cm × 3 cm rectangles and dried in an oven (Tecnal TE-393/1) at 60 ºC until constant mass. They were then placed in steel-mesh pieces to ensure contact between the film and the soil and facilitate their removal after the weighing period. The steel meshes containing the film samples were folded, placed approximately 4 cm below the container surface, and covered with soil to keep them buried. Water was added to the soil every two days to maintain soil moisture at approximately 40%. The experiment was conducted in triplicate, and the films were evaluated at 0, 5, 10, and 15 days under the previously described conditions. The films were dug up after each established period and removed from contact with the soil using tweezers. They were carefully washed with distilled water to remove traces of soil and then dried at 60 ºC in an oven until constant mass. The degree of biodegradation of the films was determined as weight loss.

Statistical analysis

The data obtained from the starch granule characterization analyses were submitted to analysis of variance (ANOVA). The films were subjected to ANOVA and; subsequently, to Dunnett's test with a 5% significance level to compare the effects of fermentation and emulsion addition on film properties relative to the Fnat control treatment (film with native starch) and Tukey's test for comparison between treatments with modified starch (Fmod, F0.2, F0.4, and F0.6).

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RESULTS AND DISCUSSION

Characterization of native and modified starch by natural fermentation

Table 1 shows the extraction yield and chemical characterization of native (Nat) and modified (Mod) starch by natural fermentation.

The extraction yield obtained in this study was higher than those reported by FREITAS & TAVARES (2005), who reported a yield of 8% for plantain (Musa AAB-Terra), and by CHÁVEZ-SALAZAR et al. (2017) (12.73%), but lower than that reported by PACHECO et al. (2025), (20.8%) and CORDEIRO et al. (2018) (19.4%), who used the same extraction. This difference is probably related to the chosen extraction method and the fruit ripening stage (Stage 1 according to the Von Loesecke scale), both of which directly influence yield. The method for extracting starch with ammonia hydroxide (NH_4OH) demonstrated the industrial potential of plantain as a starch source. In this method, natural plantain enzymes such as polygalacturonase and pectinase are activated. These enzymes catalyze the hydrolysis of bonds in the cell walls of the pulp, allowing the release of starch granules without degrading them, leading to their recovery by filtration from the residues and to the separation of the aqueous dispersion by centrifugation (SCHOCH, 1964; ZHANG et al., 2024). These values are lower than those reported for conventional starches, such as maize starch (40.0-70.0%) (PACHECO et al., 2025), likely due to the raw material's high moisture content and the presence of non-starch polysaccharides, including dietary fibers and mucilage.

The pH and titratable acidity values of native and modified starches showed significant differences ($P < 0.05$) from each other. After the natural fermentation period of 35 days, titratable acidity increased with decreasing pH, a behavior also observed by KAROW et al. (2024). Organic acids, like lactic, propionic, and butyric, produced by acid bacteria, and succinic, produced by yeast, produced during fermentation may be primarily responsible for starch acidification and the consequent reduction in pH (JOSHI et al., 2026; ZHAO et al., 2019).

The moisture content is below the limit of 15% (g/100 g) established by RDC Nº 263 (BRASIL, 2005) for both starches, not statistically different from each other. This result is expected for this type of product, particularly considering the influence of drying conditions. Moisture content is a key parameter of the structural stability and functional properties of starch-based systems. Variations in water content directly influence molecular mobility, gelatinization behavior, and retrogradation processes. Excess moisture can promote water migration and structural disruption, leading to deterioration in texture, consistency, and overall quality of starch-containing materials (LIU et al., 2022; XIE et al., 2025).

The low ash content in native and modified starches indicates the reduced presence of inorganic material. The efficiency of the washing process during the extraction stage indicates significant removal of this material, thereby improving the purity and quality of the starch (HOOVER, 2001).

The protein and lipid contents were similar for both types of starch, low as expected for the type of product under analysis and did not

Table 1 - Characterization of the physicochemical composition of green plantain (*Musa paradisiaca* L.) starch, native and modified by natural fermentation.

Parameter	----------Nat----------	----------Mod----------
Extraction yield (g/100 g)*	15.55	---
pH	7.78 ± 0.05[a]	4.77 ± 0.52[b]
Titratable acidity (g/100 g)	0.30 ± 0.05[b]	1.18 ± 0.05[a]
Moisture (%)**	11.94 ± 0.16[a]	11.93 ± 0.10[a]
Ashes (%)**	0.51 ± 0.04[a]	0.49 ± 0.23[a]
Crude protein (%)**	0.027 ± 0.001[a]	0.027 ± 0.001[a]
Total lipids (%)**	0.123 ± 0.010[a]	0.095 ± 0.001[a]
Apparent amylose (%)**	37.52 ± 3.01[a]	31.33 ± 0.48[b]

i) Nat: native starch; Mod: modified starch. *Fresh weight. **Dry weight. Equal letters on the same row do not differ from each other by the F-test at a 5% significance level.

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differ significantly ($P > 0.05$). Therefore, they do not have a significant effect on the properties of the produced films, as high levels of proteins and lipids are undesirable, as they interact with amylose through hydrogen bonding, hydrophobic interactions, and complex formation, disrupting the starch matrix and negatively affecting the structural, mechanical, and barrier properties of starch-based films. High contents of these constituents interact with the amylose molecule. These interactions can significantly alter the structural organization of the starch matrix, thereby influencing film elasticity, flexibility, barrier properties, and mechanical resistance (PELISSARI et al., 2012a; WANG et al., 2024; ZHANG et al., 2025).

The apparent amylose content decreased after the natural fermentation modification process, and the difference was statistically significant ($P < 0.05$) compared with the native starch. This decrease may be related to the action of microbial amylolytic enzymes that trigger the hydrolysis of starch, converting it into glucose or maltose, which serve as a source of nutrition for the microorganisms present in the starch and water suspension (AQUINO et al., 2016; MONTENEGRO et al., 2008). AL-ANSI et al. (2021), investigated the impact of natural fermentation on barley starch. According to the authors, differences in molecular and crystalline structures were observed during fermentation, likely due to the degradation of starch by activated enzymes. Despite the observed reduction, the values are consistent with those reported in the literature. Studies indicated that amylose levels in green banana starch typically range from approximately 20% to 40%, although values exceeding 50% have been reported depending on cultivar and processing conditions. This variability shows a wider distribution than that of conventional starches such as maize and wheat, which generally exhibit amylose contents between 20% and 30% (DIBAKOANE et al., 2024; MARTA et al., 2022; PELISSARI et al., 2012a; ZHANG et al., 2025).

Figure 1 shows the surface morphology of native and modified starch granules in micrographs. In general, starch granules varied in size and shape, including oval and rod shapes. The granules have no visible signs of deformation, cracking, or breakage. These results suggested that surface corrosion does not occur during the natural fermentation of plantain starch granules. Previous studies on naturally fermented barley (AL-ANSI et al., 2021) and wheat (ZHAO et al., 2019) starch yielded divergent results. These authors observed that starch granules fractured and distorted after 48 h of fermentation. The absence of fractures in plantain starch granules may be related to their resistance to enzyme action during fermentation (KAROW et al., 2024). Furthermore, the structure of starch granules is relatively stable and

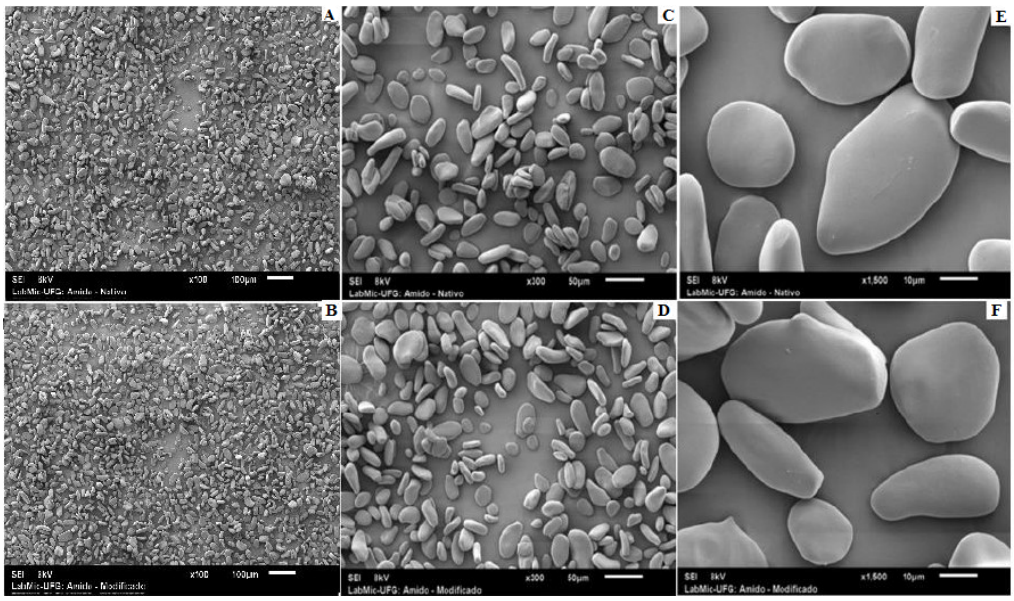


Figure 1 - Micrographs of native (A, C, E) and modified (B, D, F) starch from green plantain (*Musa paradisiaca* L.) obtained by SEM: x100 (A, B), x300 (C, D), and x1500 (E, F).

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resistant to changes induced by natural fermentation, which explains why no fractures or breaks occurred during this process. However, research on plantain starch is still in its early stages, resulting in a limited number of studies in the literature that report or explain its behavior during fermentation.

Characterization of biodegradable films

A significant difference in film thickness was observed for all treatments compared to the control (Fnat) at the 5% significance level by Dunnett's test (Table 2). This difference is related to the lower amylose content in the modified starch, which reduces film thickness by structuring the amylose-amylose network. The shrinkage of the starch gel during drying is more favored when the amylose content is lower. Amylopectin favors the production of less heterogeneous and denser films and, therefore, thinner films (BASIAK et al., 2017). However, Tukey's test at a 5% significance level showed no significant differences between treatments containing modified starch.

The water vapor permeability analysis demonstrated that the formulations containing the extract emulsion had a lower rate than the control treatment (Fnat). This reduction in permeability is attributed to several factors, including the physical barrier created by the extract, chemical interactions between the extract and the film components, and matrix compaction promoted by the addition, which reduces the available space for water molecules to pass. Thus, water molecules from outside the films could not penetrate the inter- and intramolecular spaces of the polymer chains, resulting in less moisture absorption (BHARTI et al., 2022).

Water activity analysis of the films showed no significant difference among them when maintained in a controlled water activity environment, except for the film containing 0.6% emulsion, which is probably related to its higher hydrophobicity due to the higher extract concentration. A lower water activity (values near 0.5) indicates less susceptibility of the product to microbial degradation and chemical changes.

The results of the solubility analysis showed a significant difference between the treatments (Fmod, F0.4, and F0.6) and the control (Fnat). However, the same behavior was not observed for the formulation with 0.2% emulsion (F0.2). The higher the extract concentration in the formulations, the lower their solubility capacity, due to reduced miscibility in the system. Extracts in emulsion, as complex mixtures of several chemical compounds, show that the hydrophobic character is a variable characteristic affecting the performance and barrier properties of the films (ATARÉS & CHIRALT, 2016).

Table 2 - Properties of films produced with native and modified green plantain (*Musa paradisiaca* L.) starch incorporated by natural fermentation with calendula (*Calendula officinalis* L.) extract emulsion.

Treatment	-----Fnat-----	------Fmod------	-------F0.2-------	------F0.4------	--------F0.6--------	--P-value--
Thickness	0.11 ± 0.006	0.08 ± 0.01$^{* a}$	0.1 ± 0.017$^{* a}$	0.1 ± 0.009$^{* a}$	.1 ± 0.008$^{* a}$	<.0001
WVP (g water mm·da $y^{-1}m^{-2}$ kPa^{-1})	0.37 ± 0.062	0.55 ± 0.07$^{* a}$	0.09 ± 0.02$^{* b}$	0.03 ± 0.004$^{* b}$	0.06 ± 0.006$^{* b}$	<.0001
Solubility (%)	25.40 ± 4.6	32.66 ± 5.3$^{* a}$	18.95 ± 3.30$^{ns b}$	15.27 ± 2.80$^{* b}$	15.18 ± 1.20$^{* b}$	0.0025
a_w	0.54 ± 0.02	0.53 ± 0.01$^{ns a}$	0.53 ± 0.01$^{ns a b}$	0.516 ± 0.02$^{ns a b}$	0.47 ± 0.04$^{* b}$	0.0032
Young's modulus (MPa)	69.40 ± 1.9	55.95 ± 2.7$^{* c}$	196.18 ± 6.4$^{* a}$	187.208 ± 4.1$^{* b}$	39.30 ± 3.8$^{* d}$	<.0001
Maximum stress (MPa)	10.68 ± 2.3	13.87 ± 4.1$^{ns a b}$	15.80 ± 5.5$^{* a}$	12.653 ± 2.7$^{ns a b}$	8.14 ± 2.5$^{ns c}$	<.0001
Strain (mm)	25.66 ± 3.4	29.25 ± 4.7$^{ns a b}$	21.56 ± 1.2$^{ns c}$	23.352 ± 3.6$^{ns b c}$	33.69 ± 2.6$^{ns a}$	<.0001
L^*	33.33 ± 0.9	34.64 ± 0.5$^{ns a}$	34.96 ± 1.4$^{ns a}$	33.93 ± 2.2$^{ns a}$	33.56 ± 0.8$^{ns a}$	0.1911
a^*	0.05 ± 0.06	0.08 ± 0.1$^{ns a}$	0.05 ± 0.2$^{ns a}$	0.06 ± 0.2$^{ns a}$	0.07 ± 0.1$^{ns a}$	0.9955
b^*	0.11 ± 0.09	0.20 ± 0.16$^{ns a}$	0.27 ± 0.19$^{ns a}$	0.24 ± 0.19$^{ns a}$	0.35 ± 0.1$^{* a}$	0.1367

i) Means followed by * and ^{ns} in the row differ and do not differ, respectively, from the control treatment (Fnat) by Dunnett's test at the 5% significance level. Means followed by equal letters in the column do not differ from each other for treatments (Fmod, F0.2, F0.4, and F0.6) by Tukey's test at the 5% significance.

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Food packaging plays a key role in maintaining product stability and extending shelf life. Food spoilage is closely related to environmental humidity. Biodegradable films with low permeability or barrier to water vapor and gases help maintain and extend the shelf life of packaged foods during storage (LIU et al., 2022; NI et al., 2018; PATIL et al., 2021; THAKUR et al., 2019; WANG et al., 2020).

Therefore, these results demonstrated that biodegradable films with the addition of an extract emulsion exhibit better barrier properties to water vapor, lower water activity, and lower solubility, thereby contributing to the preservation and extension of the shelf life of packaged foods during storage.

Mechanical resistance of films

Film mechanical analysis was performed by applying an axial traction force to a standardized test piece, promoting stretching until the material deformed. Table 2 presents the results of the mechanical analysis, including Young's modulus, maximum tensile strength (tensile strength), and strain at break (elongation at break).

Young's modulus, also known as the modulus of elasticity, measures the stiffness of a material in response to applying a tensile force. It describes the material's ability to deform elastically and return to its original shape. All treatments (Fmod, F0.2, F0.4, and F0.6) differed significantly from the control treatment (Fnat). Formulations containing 0.2% and 0.4% emulsion were less susceptible to deformation under external forces, indicating a more cohesive, structured polymer network. However, the elastic capacity decreased as the emulsion concentration increased to 0.6%, suggesting a reduction in matrix cohesion, as commonly reported in starch-based systems containing dispersed lipid phases (SINGH et al., 2022). This behavior can be attributed to interactions between the oil phase and the polymeric components of the film, which may interfere with intermolecular interactions and reduce chain packing efficiency. As a result, the film becomes less rigid and more susceptible to deformation (KARNWAL et al., 2025; SINGH et al., 2022). From an application standpoint, this reduction in stiffness may compromise the mechanical resistance of the film, potentially limiting its use in packaging applications requiring higher load-bearing capacity (BONILLA et al., 2012; MACHADO et al., 2020).

Maximum tensile strength is the maximum force the film can withstand before breaking during a tensile test. Only the F0.2 treatment differed statistically from the control treatment (Fnat), showing higher tensile strength. This result suggested that low emulsion concentrations (0.2%) may reinforce the polymer network, thereby improving mechanical resistance. In contrast, a higher emulsion content (0.6%) may weaken intermolecular cohesion, potentially reducing the film's ability to withstand mechanical stress.

The strain at break (elongation at break) results showed that only the F0.6 treatment differed significantly from the control (Fnat), exhibiting higher deformability. This behavior is consistent with a less cohesive, more flexible matrix, likely due to structural discontinuities introduced by the dispersed phase. While increased elongation may be advantageous for flexibility, it may also indicate reduced structural integrity, which should be considered depending on the intended application (BENAVIDES et al., 2012).

These mechanical properties are important in the packaging development process, as the material must be resistant to mechanical stress and maintain its integrity during transport, handling, and storage (RODRIGUES et al., 2021). Furthermore, the film's ability to resist deformation is directly related to its molecular structure and the presence of additives, such as the extracts used in this study. Therefore, understanding how these components affect the film's structure and mechanical properties is essential to ensure the quality and effectiveness of the packaging produced.

Structural morphology of films by scanning electron microscopy - SEM

Figure 2 shows micrographs of the films produced for structural evaluation at the surface and at fracture.

Films produced with the addition of emulsion (F0.2, F0.4, and F0.6) showed the presence of coalescences. Adding the extract emulsion at these concentrations to the matrix may have led to agglutination of oil droplets, resulting in the clots observed in the micrographs (Figures 2H, 2I, 2J). This coalescence occurs because fewer biopolymers are available for surface adsorption as extract content increases (FASIHI et al., 2023). The occurrence of superficial coalescences was more pronounced in treatment F0.4 (Figure 2I), while clots were present in treatment F0.6 (Figure 2J) throughout the matrix. This result is attributed to phase separation induced by the extract emulsion, which reduces the system's miscibility due to the presence of a more hydrophobic and a more hydrophilic phase. HAGHIGHI et al. (2019) characterized chitosan-

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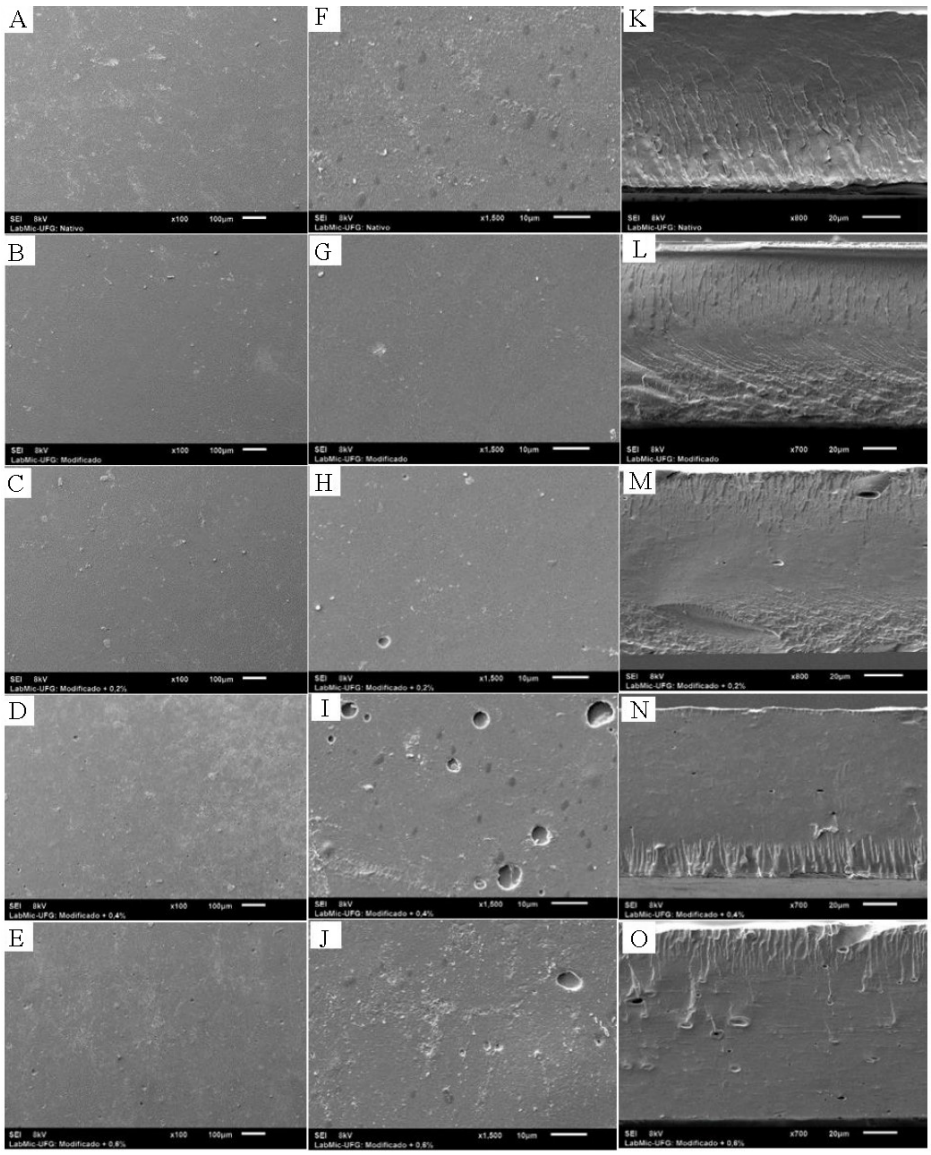


Figure 2 - Micrographs of the surface (A, B, C, D, E, F, H, I, J) and fracture (K, L, M, N, O) of the film samples of green plantain (*Musa paradisiaca* L.) starch: Fnat (A, F, K), Fmod (B, G, L), F.2 (C, H, M), F0.4 (D, I. N), and F0.6 (E, J, O) obtained by SEM: x100 (A, B, C, D, E), x1500 (F, G, H, I, J), x700 and x800 (K, L, M, N, O).

gelatin blend films enriched with different extracts and observed surface heterogeneity, attributed to droplets formed during drying.

Chemical composition by Fourier-transform infrared spectroscopy of films – FTIR

Infrared spectroscopy (FTIR) analysis using attenuated total reflectance (ATR) was performed to characterize the functional groups in the produced films (Figure 3).

The typical bands of functional groups present in starch are observed in all films. The bands corresponding to functional groups remained unchanged for the Fnat, Fmod, and F0.2 treatments, indicating that no new functional groups formed in the films under these conditions. However, films with higher concentrations of the extract emulsion (0.4% and 0.6%) showed differences in their characteristic spectra. The band at 993 cm^{-1} is associated with the amorphous structure, that is, the amount of amylose

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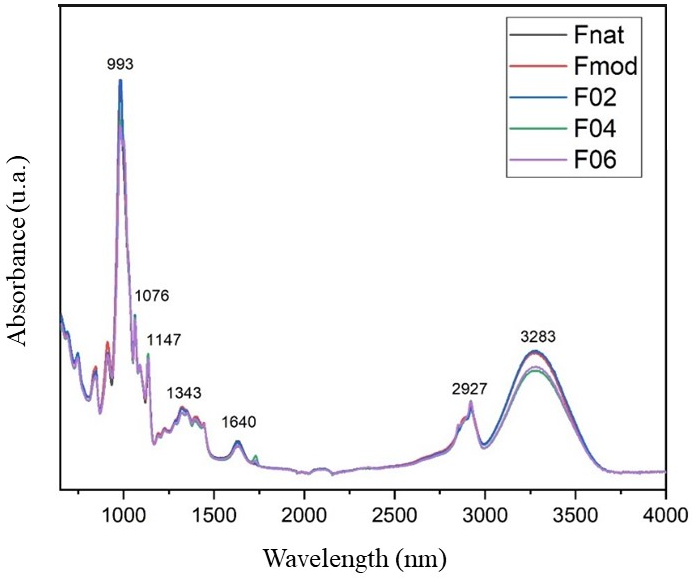


Figure 3 - ATR spectra of native and modified green plantain (*Musa paradisiaca* L.) starch films incorporated with pot marigold (*Calendula officinalis* L.) calendula extract emulsion.

in the starch sample (PELISSARI et al., 2012b). The absorption band at 1642 cm^{-1} is attributed to the intramolecular hydrogen bonding of starch (LIU et al., 2014). The small band found around 1700 cm^{-1}, observed only in film formulations containing 0.4 and 0.6% addition, corresponds to the C = O stretching vibration of calendula extract (PEDRAM RAD et al., 2019). The band located at 2927 cm^{-1} is the result of the stretching of the C-H bonds. The absorption band at 3283 cm^{-1} is associated with the stretching vibrations of the hydroxyl group (OH) present in starch (SAKKARA et al., 2020).

Optical properties

Table 2 shows the results of color analysis in the films. The evaluated parameters were L^* (lightness), in which higher L^* values indicate a lighter color, while lower values indicate a darker color; a^*, which represents the hue from green ($-a^*$) to red ($+a^*$), and b^*, which goes from blue ($-b^*$) to yellow ($+b^*$). The treatments showed no statistically significant differences relative to the control treatment for L^* and a^*. However, only the F0.6 treatment showed a significant difference for the b^* parameter compared to the control (Fnat). Therefore, the calendula extract emulsion showed the highest concentration and a more yellowish color, increasing the b^* parameter.

Determination of antioxidant capacity by the DPPH and FRAP methods

Table 3 shows the results of antioxidant capacity using the DPPH and FRAP methods. All treatments differed from the control treatment (Fnat) at the 5% significance level by Dunnett's test using the FRAP method. However, the treatments of films with modified starch showed no significant difference from each other by Tukey's test at the 5% level. Films containing 0.4 and 0.6% extract emulsion showed significant differences from the control treatment (Fnat) in antioxidant capacity as determined by the DPPH assay, indicating that the incorporation of the emulsion contributed to an increase in radical scavenging activity. Although the absolute values were low, this result should not be interpreted as an absence of antioxidant functionality, but rather as a reflection of the limited concentration of active compounds and their restricted mobility within the polymeric matrix. In contrast, the FRAP results showed no significant differences among the film formulations, with values similarly low compared to those of the isolated emulsion. This difference between DPPH and FRAP responses suggested that the antioxidant compounds present in the system may be more effective as radical scavengers than as reducing agents, highlighting the importance of using complementary assays to properly characterize

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Table 3 - Antioxidant capacity and biodegradability indices of films produced with native green plantain (*Musa paradisiaca* L.) starch and modified by natural fermentation incorporated with calendula (*Calendula officinalis* L.) extract emulsion.

Treatment	DPPH (%)	FRAP (mg Fe^{2+}/g of extract)	Biodegradability 5th day (%)	Biodegradability 10th day (%)	Biodegradability 15th day (%)
Fnat	0.32 ± 0.01	1.13 ± 0.002	34.48 ± 2.97	38.88 ± 2.24	69.1 ± 1.85
Fmod	0.63 ± 0.01$^{*\,b}$	1.09 ± 0.003$^{*\,b}$	35.87 ± 1.46$^{ns\,a}$	36.36 ± 0.90$^{ns\,b}$	57.9 ± 2.25$^{ns\,c}$
F0.2	0.32 ± 0.01$^{ns\,d}$	1.10 ± 0.003$^{*\,b}$	37.03 ± 0.83$^{ns\,a}$	36.82 ± 2.95$^{ns\,b}$	81.52 ± 3.31$^{*\,b}$
F0.4	0.47 ± 0.01$^{*\,c}$	1.11 ± 0.003$^{*\,b}$	30.85 ± 3.97$^{ns\,b}$	35.51 ± 4.73$^{ns\,b}$	100.00 ± 0.00$^{*\,a}$
F0.6	0.47 ± 0.01$^{*\,c}$	1.11 ± 0.003$^{*\,b}$	30.85 ± 3.97$^{ns\,b}$	100.00 ± 0.00$^{*\,a}$	100.00 ± 0.00$^{n*\,a}$
Emulsion	1.20 ± 0.1$^{*\,a}$	1.94 ± 0.03$^{*\,a}$	-	-	-

i) Means followed by * and ^{ns} in the row differ and do not differ, respectively, from the control treatment (Fnat) by Dunnett's test at the 5% significance level. Means followed by equal letters in the column do not differ from each other for treatments (Fmod, F0.2, F0.4, and F0.6) by Tukey's test at the 5% significance.

antioxidant behavior. Moreover, the polymeric network of the starch-based films likely limits the diffusion and accessibility of these compounds, reducing their measurable ferric-reducing capacity.

The antioxidant capacity of the isolated emulsion incorporated into the film was reduced relative to the emulsion, even at low levels. This decrease can be attributed to a possible chemical interaction between the extract emulsion and the matrix polymer, leading to a reduction in antioxidant capacity. Furthermore, the production process of biodegradable films can compromise the stability of the antioxidant agent, as elevated drying temperatures can degrade it.

Although studies have reported the antioxidant potential of *Calendula officinalis* L. (AK et al., 2020; ERCETIN et al., 2012; PREETHI et al., 2006; WAWOCZNY et al., 2025), the results obtained in this study indicate that, under the conditions evaluated, the incorporation of the essential oil in emulsion form at fixed and relatively low concentrations limits its detectable antioxidant capacity in the films. Nevertheless, the observed increase in DPPH activity demonstrates that the system retains some functional antioxidant capacity, which may still be important for applications where moderate oxidative protection is sufficient.

Biodegradability analysis of films

Table 3 shows the results of the biodegradability analysis of films. The quantitative index used was weight loss, in which the F0.6 treatment demonstrated a high biodegradability rate at the end of the 5th day, differing from the control (Fnat), and continuing until the 10th day, with the highest degradation rate. Despite the lower emulsion solubility, which can normally delay film degradation, other factors may have a more significant impact; for example, structural collapse may occur upon incorporation of the emulsion into the matrix, as evidenced by SEM micrographs showing increasing coalescence with rising emulsion concentration. Therefore, films with added extract emulsion may degrade more quickly, even with this limited solubility, due to a combination of biological and structural factors. Biological degradation of polymers refers to any modification of the polymeric material's characteristics, including decomposition by microbial enzymes, a decrease in molecular weight, and deterioration of mechanical strength and surface properties. It can be defined as the fragmentation of material by digesting microorganisms (PATHAK & NAVNEET, 2017). Polymer structures play a key role in determining the degradation rate. Surface conditions, such as hydrophilicity and surface area, are directly related to a material's overall degradability. In addition, external environmental factors, such as humidity and temperature, also have an influence. The presence of moisture introduces water molecules into the polymer, which can trigger hydrolysis depending on the polymeric material's susceptibility or hydrophobicity. The polymer crystallinity is closely related to its degradation. Thus, the lower the degree of crystallinity, the higher the polymer degradability (ATTALLAH et al., 2021). The periodic addition of water may also have contributed to the loss of soluble compounds, leading to a loss of initial appearance and structural integrity and to significant degradation by the end of the 15th day of evaluation.

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CONCLUSION

Analyses of the chemical and physical properties of native and modified starches showed that the fermentation process did not affect the granular structure of the starch, although it did alter apparent amylose, pH, and acidity. The addition of extract emulsion improved water vapor permeability resistance but negatively impacted some mechanical properties, such as the reduction in the modulus of elasticity. The antioxidant capacity of the films was not considered low, despite the low concentration of extract in the emulsion. An increase in biodegradability was observed with increasing emulsion concentration in the formulations. This behavior indicates that the incorporation of the emulsion, particularly at the F0.6 level, effectively enhances the biodegradation kinetics of the films, promoting faster initiation and a greater extent of degradation compared to the control, which highlights its potential for developing more environmentally responsive biodegradable materials. These results demonstrated the potential of green plantain starch-based biodegradable films as a sustainable alternative to conventional plastic packaging. However, further studies are needed to better understand their behavior and to explore practical applications, including analyses of the starch viscoelastic profile, thermal properties, and microbial activity.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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DECLARATION OF CONFLICT OF INTEREST

The authors state that they do not have any competing financial interests or personal relationships that could have influenced the work reported in this paper.

AUTHORS' CONTRIBUTIONS

ELSS: Conceptualization, Methodology, Investigation, Writing – original draft, Formal analysis, Data curation. EBMV: Methodology, Investigation. LSS: Supervision. RCFB: Supervision. MPFS: Visualization, Writing – review & editing CMV: Conceptualization, Methodology, Writing – review & editing, Supervision, Formal analysis, Data curation.

DECLARATION OF USE OF ARTIFICIAL INTELLIGENCE

During the preparation of this work, the authors did not use any artificial intelligence tools.

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