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# Biodegradable bioactive films based on sorghum starch and glycerol incorporated with jabuticaba (*Plinia cauliflora*) and jambolan (*Syzygium cumini*) peels extracts

Vinícius Tadeu da Veiga Correia [a,b,*], Nayana Hayss Araújo Silva [b], Pãmella Fronza [c], Ana Luiza Santos Vieira [b], Ana Luiza Mendes Nunes da Silva [b], Isabella Maciel Costa [d], Taynan Jonatha Neves Costa [b], Bruna Maria Salotti de Souza [d], Valéria Aparecida Vieira Queiroz [e], Washington Azevedo da Silva [f], Julio Onesio-Ferreira Melo [g], Camila Argenta Fante [b]

[a] Empresa de Pesquisa Agropecuária de Minas Gerais (EPAMIG), Campo Experimental Risoleta Neves, São João del-Rei, Minas Gerais, 36301-360, Brazil
[b] Department of Food, Faculty of Pharmacy, Federal University of Minas Gerais (UFMG), Campus Pampulha, Belo Horizonte, Minas Gerais, 31270010, Brazil
[c] Food and Nutrition Institute, Federal University of Rio de Janeiro (UFRJ), Macaé, Rio de Janeiro, 27930-560, Brazil
[d] Department of Technology and Inspection of Animal Products, Veterinary School, Federal University of Minas Gerais (UFMG), Belo Horizonte, 31270-901, Brazil
[e] Embrapa Milho e Sorgo, Sete Lagoas, Minas Gerais, 35701-970, Brazil
[f] Department of Food Engineering, Federal University of São João del-Rei (UFSJ), Campus Sete Lagoas, Sete Lagoas, MG, 35702-031, Brazil
[g] Department of Exact and Biological Sciences, Federal University of São João del-Rei (UFSJ), Campus Sete Lagoas, Sete Lagoas, MG, 35702-031, Brazil

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ABSTRACT

Growing interest in renewable and biodegradable resources has encouraged industries to reduce reliance on petrochemical-based packaging. As an alternative, films developed from unconventional starch sources and fruit by-products have been explored. This study aimed to produce films from sorghum starch combined with jabuticaba and jambolan extracts from freeze-dried peels and to evaluate their physical, barrier, optical, thermal, mechanical, and biodegradation properties. Films prepared with sorghum starch were supplemented with glycerol and incorporated with jabuticaba (JT) or jambolan (JB) freeze-dried peels extracts. The formulations included control film (without extract) and films with added extract at concentrations of 5% (JT5% or JB5%) and 10% (JT10% or JB10%). Anthocyanin levels above 4.40 and 7.29 mg C3Geq 100 $g^{-1}$ were detected in JT5% and JT10%, respectively. The highest luminosity (L* = 88.82) was observed in the control film. Extract-enriched films showed opacity values above 1.7. JT10% exhibited the greatest thickness (0.17 mm), while JB10% had the highest water solubility (22.36%). JT5% displayed the highest water vapor permeability (2.09 g·mm/h·$m^2$·kPa). Extract addition did not influence puncture resistance, however, tensile strength decreased, with JB10% showing the lowest value (1.65 MPa). JT10% demonstrated the greatest flexibility, with 26.86% elongation at break. Scanning electron microscopy revealed no cracks but a more homogeneous surface in the control film. All films exhibited characteristic bands of starch–glycerol thermoplastics and fully degraded within 7 days. Overall, the films developed exhibited a combination of adequate mechanical performance, modified optical, and rapid biodegradation, supporting their potential use as materials for packaging food applications.

## 1. Introduction

The excessive use of single-use plastic products has led to serious environmental issues, including air, soil, and water pollution, mainly due to inadequate waste management, improper disposal practices, and their long persistence in the environment [1]. In this context, increasing environmental awareness has intensified the search for renewable and biodegradable alternatives, particularly for packaging applications [2].

* Corresponding author at: Empresa de Pesquisa Agropecuária de Minas Gerais (EPAMIG), Campo Experimental Risoleta Neves, São João del-Rei, Minas Gerais, 36301-360, Brazil.
*E-mail address:* vinicius.correia@epamig.br (V.T. da Veiga Correia).

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Among the available biodegradable materials, films have emerged as a promising strategy to reduce dependence on petrochemical-based packaging [3].

According to Shahidi and Hossain [4], films are thin layers used as primary food packaging, produced through processes such as casting, extrusion, dip coating, and spin coating, and subsequently applied to food surfaces, between food components, or as sealing materials. Their main function is to protect foods against mechanical, oxidative, and microbiological damage [5]. Natural macromolecules with film-forming properties, especially proteins and polysaccharides, have been widely investigated for this purpose. Among these materials, starch stands out due to its neutral sensory characteristics, low cost, wide availability, and good film-forming ability [6,7].

In recent years, growing attention has been directed toward unconventional starch sources, aiming to expand the range of sustainable raw materials for biodegradable films. Sorghum, the fifth most produced cereal worldwide, has gained interest due to its agronomic advantages, such as low production costs and high resilience, particularly in developing countries [8–10]. Sorghum starch (BR501) presents a homogeneous matrix with approximately 28.7% amylose, a content higher than that of commercial corn starch, which favors the formation of stable and cohesive film structures [10]. Although previous studies have reported the development of sorghum starch-based biodegradable films, the incorporation of functional additives into this matrix remains limited and deserves further investigation [10,11].

Fruits and vegetables are recognized as important sources of bioactive compounds with antioxidant properties, distributed among different plant fractions such as pulps, seeds, leaves, and peels [12]. Fruit peels, often discarded as agro-industrial waste, are particularly rich in phenolic compounds, including tannins, flavonoids, and anthocyanins, as well as dietary fibers and other polyphenols. Beyond their antioxidant capacity, these compounds can interact with polymeric matrices, potentially modifying the mechanical, structural, and functional properties of biodegradable films. Therefore, the valorization of agro-industrial by-products represents a sustainable strategy to reduce waste, add economic value, and develop functional materials for food packaging applications [3].

Although several plant extracts have been investigated for incorporation into polymeric materials, jabuticaba and jambolan peel extracts deserve particular attention due to their high anthocyanin and phenolic contents, mainly associated with compounds such as cyanidin-3-glucoside, delphinidin derivatives, ellagic acid, gallic acid, and other flavonoids, which are responsible for their high antioxidant capacity [13]. Anthocyanins, which are water-soluble pigments responsible for the red and purple coloration of fruits, exhibit pronounced antioxidant activity and sensitivity to pH changes and metal complexation, making them promising functional additives for biodegradable packaging systems [14].

In addition, the phenolic compounds may interact with starch chains through hydrogen bonding, acting as secondary plasticizers and contributing to improvements in film flexibility and overall mechanical performance, which are typically limited in starch-based materials [15]. However, studies addressing the interaction of these extracts with unconventional starch matrices, such as sorghum starch, and their effects on the properties of films remain limited [10,11,16].

Thus, it was hypothesized that the incorporation of anthocyanin-rich jabuticaba and jambolan peel extracts into sorghum starch films could modulate their functional properties, particularly optical behavior and antioxidant potential, while maintaining suitable physicochemical performance. In this context, this study aimed to investigate the effect of adding jabuticaba or jambolan peel extracts to a sorghum starch film matrix for the development of potentially active films.

## 2. Material and methods

### 2.1. Material

Ripe fruits of jabuticaba and jambolan were collected in the city of Paraopeba, Minas Gerais, Brazil (19°16′54″ S, 44°24′32″ W, and 741 m). After harvesting, the fruits were washed, sanitized with a 200 mg·L$^{-1}$ solution, and peeled. The peels were then freeze-dried for 72 h in a freeze dryer (L101 LIOTOP – LIOBRAS). Freeze-dried peels (1.0 g, of each sample separately) were mixed with 10 mL of acidified water (pH 4.5, adjusted with citric acid), vortexed for 30 s, and centrifuged at 1370 $\times g$ for 15 min. The supernatants were transferred to tubes wrapped in aluminum foil to prevent photodegradation.

For the preparation of biodegradable films, sorghum starch from the BR 501 genotype (white pericarp and tannin-free), supplied by Embrapa Milho e Sorgo, was used as the film-forming matrix. The starch contained 9.08 ± 0.22% moisture, 0.89 ± 0.12% protein, 0.36 ± 0.13% lipids, 1.04 ± 0.15% ash, and 88.63 ± 0.16% carbohydrates, with an amylose content of 28.7 ± 0.4% [10]. Glycerol (Dinâmica Química Contemporânea LTDA, São Paulo, Brazil) was employed as the plasticizer. All other reagents used for the analyses were of analytical grade.

### 2.2. pH sensitivity, total anthocyanins, total phenolic content and antioxidant capacity of the extracts

The pH sensitivity of jabuticaba (JT) and jambolan (JB) extracts was evaluated by adding 3 mL of each extract separately to 25 mL of buffer solutions covering a pH range from 2 to 11 [17]. To determine the total anthocyanin content, the method proposed by Francis [18] was applied, using a spectrophotometer (FEMTO, Cirrus 80) at 535 nm. Results were expressed as mg of cyanidin-3-glucoside per 100 g of sample. The total phenolic compound content of the extracts was determined using the Folin–Ciocalteu reagent method, and the results were expressed as gallic acid equivalents (mg GAE·100 g$^{-1}$ sample). Antioxidant capacity was evaluated by spectrophotometry using three complementary assays: DPPH (2,2-diphenyl-1-picrylhydrazyl) at 515 nm [19], FRAP (ferric reducing antioxidant power) at 595 nm [20], and ABTS [2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid)] at 734 nm [21].

### 2.3. Elaboration of filmogenic suspension

The filmogenic suspension was prepared by dispersing sorghum starch in distilled water (4% w/w), as reported in Correia et al. [10], and heating it in a water bath (GMA Medical™; Brazil) to 90 °C under constant agitation. Glycerol was then added (40% w/w relative to sorghum starch), and the mixture was heated for 30 min. After cooling the filmogenic solution to 45 °C, extracts of jabuticaba or jambolan freeze-dried peels were incorporated according to the amount of filmogenic solution obtained, at the following concentrations: 5% (JT5%) and 10% (JT10%) w/w of jabuticaba extract, and 5% (JB5%) and 10% (JB10%) w/w of jambolan extract. Aliquots of 60 g of the filmogenic solution were poured into polystyrene Petri dishes (15 cm diameter) and dried in a forced-air oven (Fanem, Model 320-SE; São Paulo, Brazil) at 30 °C for approximately 18 h. The films were placed in desiccators containing saturated sodium bromide solutions to maintain a relative humidity of 58% at 25 °C for 48 h prior to characterization [15].

The glycerol concentration (40% w/w, relative to starch) was defined based on the findings of Correia et al. [10], who employed this plasticizer concentration. The concentrations of 5% and 10% (w/w, relative to the film-forming solution) were selected based on previous studies reporting the incorporation of plant extracts into starch-based films within this range [15,22], allowing the evaluation of concentration-dependent effects while maintaining film-forming ability and structural integrity, as confirmed through preliminary tests.

### 2.4. Characterization of the films

#### 2.4.1. General analysis

Visual and tactile analyses were performed to assess the homogeneity and flexibility of the films, as well as to detect the presence of cracks and/or bubbles [23].

#### 2.4.2. Total anthocyanins contents, total phenolic content and antioxidant capacity of the films

To determine the total anthocyanin content, total phenolic content and antioxidant capacity of the films, the methods described in Section 2.2 were used [18–21].

#### 2.4.3. Color determination, opacity and moisture content of films

A digital colorimeter (model CM-2600D, Konica Minolta, Osaka, Japan) was employed to evaluate color parameters based on the CIELAB system, generating L*, a*, and b* values. From these values, the hue angle (h°, in degrees), chroma (c*, representing color intensity), and the total color difference (ΔE*) of the films containing extracts were calculated in comparison with the standard film (control) using Eq. (1):

$$\Delta E^* = \sqrt{(L^*sample - L^*control)^2 + (a^*sample - a^*control)^2 + (b^*sample - b^*control)^2} \quad (1)$$

Opacity measurements were performed according to the methodology described by Souza et al. [24]. In short, film samples (9 mm × 40 mm) were cut and placed directly into a quartz cuvette inserted into a spectrophotometer. Absorbance was recorded at 600 nm in triplicate using a UV/VIS spectrophotometer (Cirrus 80, Femto Indústria de Comércio de Instrumentos, São Paulo, SP, Brazil), with an empty cuvette as reference. Opacity was calculated as the ratio between absorbance and film thickness (mm). Moisture content was assessed by drying the films in a forced-air circulation oven at 105 °C until constant weight [25].

#### 2.4.4. Evaluation of thickness, water activity, water solubility, water vapor permeability

The thickness of the films was measured using a digital micrometer (Digimess Model, Electronic Outside Micrometer, São Paulo), with ten measurements taken at different points of each sample, and an accuracy of ±0.001 mm [26]. Another parameter evaluated was the water activity was determined by direct measurement using an AquaLab Lite 2 T analyzer (Decagon Devices Inc., Pullman, WA, USA) at 25 °C, and values were expressed in decimals. Solubility, expressed as a percentage (%), was determined following the method described by Pelissari et al. [27]. Water vapor permeability (WVP) was evaluated using the standard wet cup method established by the American Society for Testing and Materials (ASTM E96/E96M-16) [28] through gravimetric analysis.

#### 2.4.5. Scanning electron microscopy (SEM) of the films

The surface morphology of the films was analyzed by scanning electron microscopy (FEI Quanta 200 FEG). The samples were fixed on aluminum stubs with double-sided carbon tape, sputter-coated with a thin layer of gold, and observed under SEM at 100× magnification with an accelerating voltage of 30.0 kV.

#### 2.4.6. Mechanical properties: tensile and puncture tests

Tensile strength (tensile strength and elongation at break) and puncture (puncture resistance and puncture deformation) tests were performed on all films using a TAXTPLUS texture analyzer (Stable Micro Systems, Surrey, England) at a test speed of 1 mm/s. Tensile tests followed the standard method D882-12 [29], employing a 5 kg load cell and an A/TG tension gripper system. Film samples were cut into strips measuring 15 mm in width and 100 mm in length and mounted between the tension grips. For puncture tests, films were cut with scissors into circular specimens (40 mm in diameter) and tested using a cylindrical probe (4 mm in diameter), with an initial distance of 30 mm between the probe and the sample. Mechanical properties were evaluated in triplicate, and each replicate consisted of five measurements for both tests.

#### 2.4.7. FTIR and thermogravimetry (TGA)

Fourier transform infrared spectrophotometer (FTIR) (IRAffinity-1, Shimadzu, Japan) was used to obtain the FTIR spectra of the films in the scanning range of 4000–400 $cm^{-1}$. For the thermogravimetric (TG) analysis, approximately 2.0 mg of each sample was placed in aluminum crucibles under synthetic air atmosphere (50 $mL \cdot min^{-1}$), with a heating rate of 10 $°C \cdot min^{-1}$, in the temperature range of 30–600 °C. These conditions were applied to evaluate the thermal stability and degradation behavior of the materials [30].

#### 2.4.8. Biodegradation

The biodegradation of the films was evaluated using a soil burial method, as described by Boeira et al. [31], with modifications. The films were cut into 40 × 40 mm pieces, weighed, and placed in nylon mesh bags. The samples were then buried in plastic containers filled with natural soil, at a depth of 4 cm from both the bottom and the surface of the container. To simulate natural conditions, 20 mL of water were added daily to maintain soil moisture. Additionally, the weights of the samples were recorded, and photographs were taken to construct a degradation kinetics graph and to visually assess the decomposition of the films.

### 2.5. Statistical analysis

Results are presented as means ± standard deviation. One-way analysis of variance (ANOVA) was performed using Statistical Package for the Social Sciences (SPSS) 15.0 for Windows (SPSS Inc., Chicago, IL, USA), followed by Tukey's test for multiple comparisons, with a significant level of 5%.

## 3. Results and discussion

### 3.1. Effect of pH on the stability and bioactivity of extracts

The color of the jabuticaba and jambolan extracts varied according to the pH conditions (Fig. 1). At acidic pH values, jabuticaba extracts gradually shifted from red to reddish-brown, whereas jambolan extracts changed from intense red/pink to light purple shades. Under alkaline pH, the color of jabuticaba extracts ranged from dark brown to green, while jambolan extracts shifted from light blue to yellow (Fig. 1).

The color variations observed may be attributed to the specific types of anthocyanins present in the samples [32]. The differences in color responses observed for jabuticaba and jambolan peels extracts within the same pH range can be mainly attributed to differences in the composition of their major anthocyanins. While jabuticaba is predominantly rich in cyanidin monoglycosides, jambolan presents a high concentration of delphinidin and malvidin diglycosides. These structural variations, related to the type of aglycone and the glycosylation pattern,

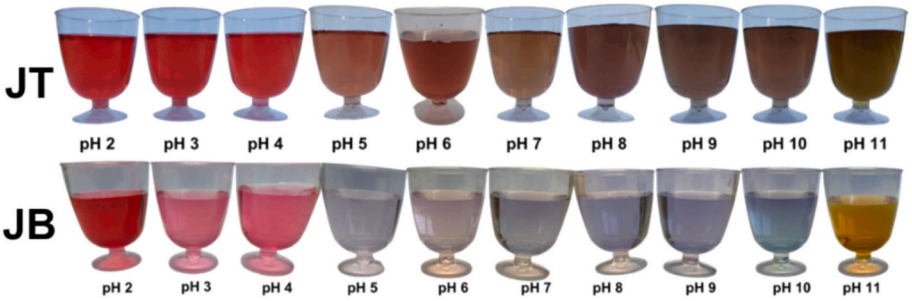


**Fig. 1.** Color variations of jabuticaba (JT) and jambolan (JB) extracts in different pH solutions.

influence the stability of the different molecular forms of anthocyanins and, consequently, the chromatic behavior of the extracts in response to pH variations [12,33].

These changes result from structural transformations of anthocyanin molecules, which can exist in different forms: the flavylium cation (red), the quinoidal base (purple to blue), the carbinol pseudo-basic (colorless), and chalcone (yellow) [34]. Therefore, the incorporation of anthocyanin-rich extracts into films can serve as a natural pH indicator, enabling the monitoring of food quality and providing insights into product deterioration during storage.

At pH values ranging from 2.0 to 3.0, the red coloration is typically attributed to the transformation of the anthocyanin structure into the flavylium cation [35] under acidic conditions (Fig. 1 – JT and JB). In the pH range between approximately 5 and 7, anthocyanins undergo significant structural changes, in which nucleophilic hydration and deprotonation of the flavylium cation are the predominant processes [36]. In the jambolan extract (Fig. 1 – JB), the grayish-purple hue observed at pH 7.0, 8.0, and 9.0, as well as the blue coloration at pH 10, may be associated with the conversion of anthocyanins into alkaline quinoidal structures. The intense yellow color detected at pH 11 is likely due to the structural rearrangement into the chalcone form [37].

The development of films capable of indicating product status to consumers represents valuable technology, particularly for applications in products with reduced preservative content [38]. While such films may help reduce environmental pollution, they can also interact with both the food matrix and the surrounding environment [39].

Bioactive films rich in anthocyanins may undergo changes in the chemical state of these compounds during storage due to variations in the polymer matrix microenvironment, which may result in color changes [14]. Similarly, exposure to alkaline conditions or volatile nitrogen compounds, such as ammonia, can induce structural changes in anthocyanins, reflected as color variations [40]. Although this colorimetric response was not evaluated in the present study, these characteristics indicate promising potential for future investigations aimed at developing films for application as colorimetric indicators in intelligent packaging systems.

The extract obtained from jabuticaba peel exhibited a total anthocyanin content of 1254.70 ± 3.81 mg C3G eq. 100 $g^{-1}$ of sample, whereas the jambolan peel extract showed 971.93 ± 2.45 mg C3G eq. 100 $g^{-1}$. Both agro-industrial residues presented substantially higher anthocyanin concentrations than those reported for other plant by-products, such as winemaking grape pomace (149 mg 100 $g^{-1}$) [41], as well as other plant sources, for example black rice (*Oryza sativa* L.), which contains approximately 56.8 mg 100 $g^{-1}$ [42].

The results for total phenolic content and antioxidant capacity of the extracts, as determined by the ABTS, FRAP, and DPPH assays, are presented in Table 1. Jabuticaba peel extracts exhibited higher value than jambolan peel extracts for all evaluated bioactive parameters ($p < 0.05$). According to Prakruthi et al. [43], the observed concentrations of these compounds contribute to the coloration of the extract and to its bioactive properties, indicating its potential application in the food, nutraceutical, and cosmetic sectors. In the present study, the direct incorporation of the extract into the film-forming solution favors the distribution of these bioactive compounds within the polymeric matrix, which indicates the potential application of the films in food systems and other matrices of interest.

**Table 1**
Total phenolic compounds and antioxidant capacity of jabuticaba and jambolan peel extracts.

| | Total phenolics (mg GAE·$g^{-1}$) | ABTS (μM Trolox·$g^{-1}$) | FRAP (μM FeSO4·$g^{-1}$) | DPPH (μM Trolox·$g^{-1}$) |
|---|---|---|---|---|
| JT | 1466.47 ± 79.90[a] | 223.10 ± 11.07[a] | 417.35 ± 3.04[a] | 328.59 ± 17.99[a] |
| JB | 996.59 ± 69.77[b] | 194.51 ± 13.48[b] | 395.27 ± 2.81[b] | 255.23 ± 21.07[b] |

Means followed by the same letter in the same column do not differ significantly at the 5% by Tukey's test. JT: jabuticaba peel extract; JB: jambolan peel extract.

### 3.2. General aspects

In general, all films exhibited no visible cracks or bubbles and could be easily removed from the support plates without tearing after drying. As shown in Fig. 2, the films were homogeneous, and handling demonstrated that they were neither sticky nor brittle.

### 3.3. Anthocyanins, total phenolic contents, antioxidant capacity and optical properties of films

The total anthocyanin content in the films was 0.07 ± 0.02 mg c3g eq. 100 $g^{-1}$ for the control formulation, 1.24 ± 0.05 and 2.98 ± 0.03 mg c3g eq. 100 $g^{-1}$ for the JB5% and JB10% films, respectively, and 4.41 ± 0.10 and 7.29 ± 0.53 mg c3g eq. 100 $g^{-1}$ for the JT5% and JT10% films. The anthocyanin content in films produced with jabuticaba peel extracts was significantly higher than in those containing jambolan peel extracts ($p < 0.05$). Moreover, both extract-based films exhibited significantly higher anthocyanin levels compared to the control. As expected, the films that incorporated 10% extracts, either jabuticaba or jambolan, presented anthocyanin concentrations 1.74 (JB5% and JB10%) and 2.88 (JT5% and JT10%) mg c3g eq. 100 $g^{-1}$ higher than their 5% equivalents ($p < 0.05$).

The total phenolic content and antioxidant capacity of the developed films are presented in Table 2. A significant increase ($p < 0.05$) in total phenolic content was observed as the concentration of the incorporated extracts increased, regardless of the plant source (jabuticaba or jambolan peels). These findings are consistent with previous studies reporting higher total phenolic contents in starch-based films and enhanced antioxidant capacity with increasing concentrations of plant extracts [15,22].

The extracts were incorporated into sorghum starch–glycerol films to impart antioxidant capacity to the material. In all antioxidant assays performed (ABTS, FRAP, and DPPH), the JT10% film exhibited the highest values, followed by JT5% and JB10%, then JB5%, while the control film (without extract addition) showed non-quantifiable results.

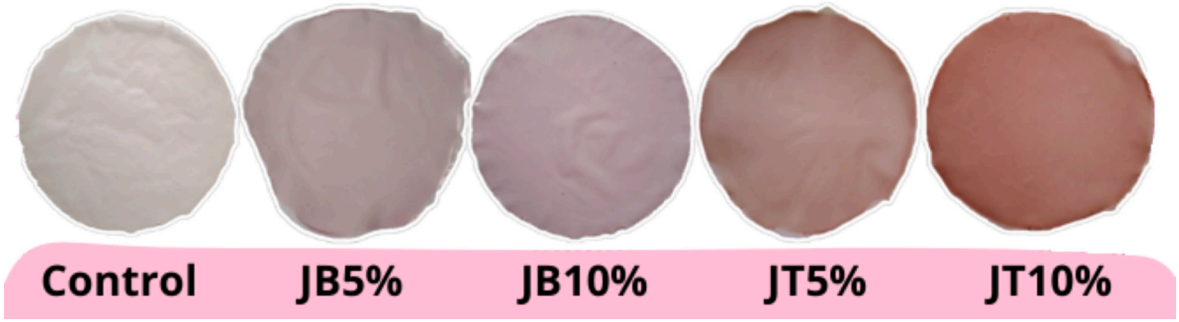


**Fig. 2.** Sorghum starch and glycerol films added with aqueous extracts of freeze-dried jabuticaba or jambolan peels. Control: film without added extract, JB5% and JB10% films with added jambolan extracts, JT5% and JT10% films with added jabuticaba extracts.

**Table 2**
Total phenolic compounds and antioxidant capacity of the films.

| Films* | Total phenolics (mg GAE·g$^{-1}$) | ABTS (μM Trolox·g$^{-1}$) | FRAP (μM FeSO4·g$^{-1}$) | DPPH (μM Trolox·g$^{-1}$) |
|---|---|---|---|---|
| Control | - | - | - | - |
| JT5% | 6.23 ± 0.72$^{b}$ | 47.19 ± 2.43$^{b}$ | 176,17 ± 7.22$^{b}$ | 41.33 ± 2.21$^{b}$ |
| JT10% | 9.17 ± 0.24$^{a}$ | 64.03 ± 3.04$^{a}$ | 214.84 ± 5.31$^{a}$ | 62.48 ± 4.37$^{a}$ |
| JB5% | 3.08 ± 0.91$^{c}$ | 22.27 ± 1.18$^{c}$ | 120.10 ± 8.93$^{c}$ | 32.01 ± 1.32$^{c}$ |
| JB10% | 6.91 ± 0.15$^{b}$ | 46.25 ± 1.14$^{b}$ | 174.23 ± 5.87$^{b}$ | 42.67 ± 1.48$^{b}$ |

- undetected. Means followed by the same letters in the same column do not differ significantly at 5% by Tukey's test.
* Control (film without addition of extract), JT5% and JT10% (Films with addition of 5% and 10% of jabuticaba extract respectively), JB5% and JB10% (Films with addition of 5% and 10% of jambolan extract respectively).

The antioxidant capacity of phenolic compounds is strongly influenced by their chemical structure, which is mainly associated with the ability of the phenolic ring to stabilize and delocalize unpaired electrons [44]. This activity is affected by the number and position of hydroxyl groups within the molecular structure and is therefore closely related to the degree of hydroxyl substitution [45].

In this context, Cao, Sofic, and Prior [46] report that flavonoids exhibit higher antioxidant capacity compared to other classes of phenolic compounds. Thus, the differences observed in total phenolic content, anthocyanin concentration, and antioxidant activity among the films can be attributed to variations in the phenolic composition of the extracts and their interactions with the polymeric matrix. Extracts richer in anthocyanins, particularly those with higher degrees of hydroxylation, can contribute more effectively to radical scavenging activity, which was reflected in the antioxidant performance of the films.

According to Singh, Kim and Lee [47], phenolic compounds and, consequently, their antioxidant capacity often exhibit properties that prevent oxidative reactions and contribute to antimicrobial effects in foods. Flavonoids, tannins, and phenolic acids present in jabuticaba and jambolan peels have shown promise as functional additives in active packaging systems and edible films or coatings. Moreover, these compounds can help maintain the physicochemical stability of food products, preserve sensory attributes, and protect foods against oxidative deterioration [12,14,47].

**Table 3**
Colorimetric properties of films based on sorghum starch and glycerol developed with or without jabuticaba or jambolan extract.

| Films* | L* | C* | H° | ΔE* |
|---|---|---|---|---|
| Control | 88.82 ± 0.48$^{a}$ | 6.61 ± 0.76$^{d}$ | 290.78 ± 1.97$^{c}$ | – |
| JT5% | 75.68 ± 0.89$^{d}$ | 7.89 ± 0.47$^{c}$ | 10.92 ± 3.45$^{e}$ | 16.18 ± 1.35$^{b}$ |
| JT10% | 67.73 ± 1.65$^{e}$ | 15.44 ± 1.25$^{a}$ | 30.36 ± 2.29$^{d}$ | 27.62 ± 2.09$^{a}$ |
| JB5% | 83.72 ± 0.36$^{b}$ | 7.90 ± 0.33$^{c}$ | 312.56 ± 1.76$^{b}$ | 6.00 ± 0.62$^{d}$ |
| JB10% | 79.34 ± 1.41$^{c}$ | 10.48 ± 0.47$^{b}$ | 317.48 ± 5.07$^{a}$ | 11.02 ± 1.40$^{c}$ |

Means followed by the same letters in the same column do not differ significantly at 5% by Tukey's test.
* Control (film without addition of extract), JT5% and JT10% (Films with addition of 5% and 10% of jabuticaba extract respectively), JB5% and JB10% (Films with addition of 5% and 10% of jambolan extract respectively).

Unlike the control film, which was transparent with a whitish appearance, the films containing extracts exhibited distinct coloration, red or purple, corresponding to the hues of jabuticaba and jambolan freeze-dried peels, respectively (Table 3).

Increasing the concentration of extracts (5 or 10% JB–JT) incorporated into the sorghum starch–glycerol films resulted in significant differences ($p < 0.05$) in C* values and total color difference (ΔE*). In addition, L* decreased as higher levels of extracts were added to the formulations, reaching the lowest value at JT10% (67.73). The highest L* was identified in the control film (88.82). Films containing jabuticaba extracts were darker than those prepared with jambolan extracts ($p < 0.05$).

Anthocyanins are pigments responsible for the red to purple hues of plant tissues and are present in both jabuticaba and jambolan peels, as reported by Correia et al. [12]. These compounds, synthesized through the plant secondary metabolism, mainly influenced the Hue angle (h°) of the developed materials. Values close to 0° (as observed for JT5% and JT10%) indicate a tendency toward red coloration, while angles above 300° correspond to purple or reddish-purple colors, as verified in the films containing jambolan extracts [48].

In terms of ΔE, Moura et al. [49] reported that values between 0.2 and 0.5 correspond to color differences with minimal visual perception. In contrast, values above 6.0 (Table 3), indicate a strong visual difference, consistent with human eye perception. The opacity values of the films were 1.55 ± 0.27 for the control, 2.02 ± 0.13 for JT5%, 2.12 ± 0.25 for JT10%, 1.70 ± 0.03 for JB5%, and 1.73 ± 0.09 for JB10%. Films containing jabuticaba extracts differed from the other samples ($p < 0.05$), whereas those incorporating jambolan extracts were similar to the control ($p > 0.05$).

Considering the application of the developed materials as packaging for oxidizable foods, the opacity of films containing jabuticaba peel extracts may be advantageous. According to Luz et al. [50], this effect results from the selective light absorption of polyphenols present in these extracts. Light penetrating the packaging material can induce oxidation reactions in foods by interacting with photosensitive compounds, thereby reducing their shelf life [51].

According to Nowak et al. [51], the primary function of packaging is to protect products against adverse effects of ultraviolet light, among other factors, and to ensure that foods maintain their sensory attributes such as color and flavor, as well as their nutritional quality. These authors also emphasize that the use of natural dyes can reduce the reliance on synthetic colorants in the food industry.

In addition to increased opacity, films containing jabuticaba peel

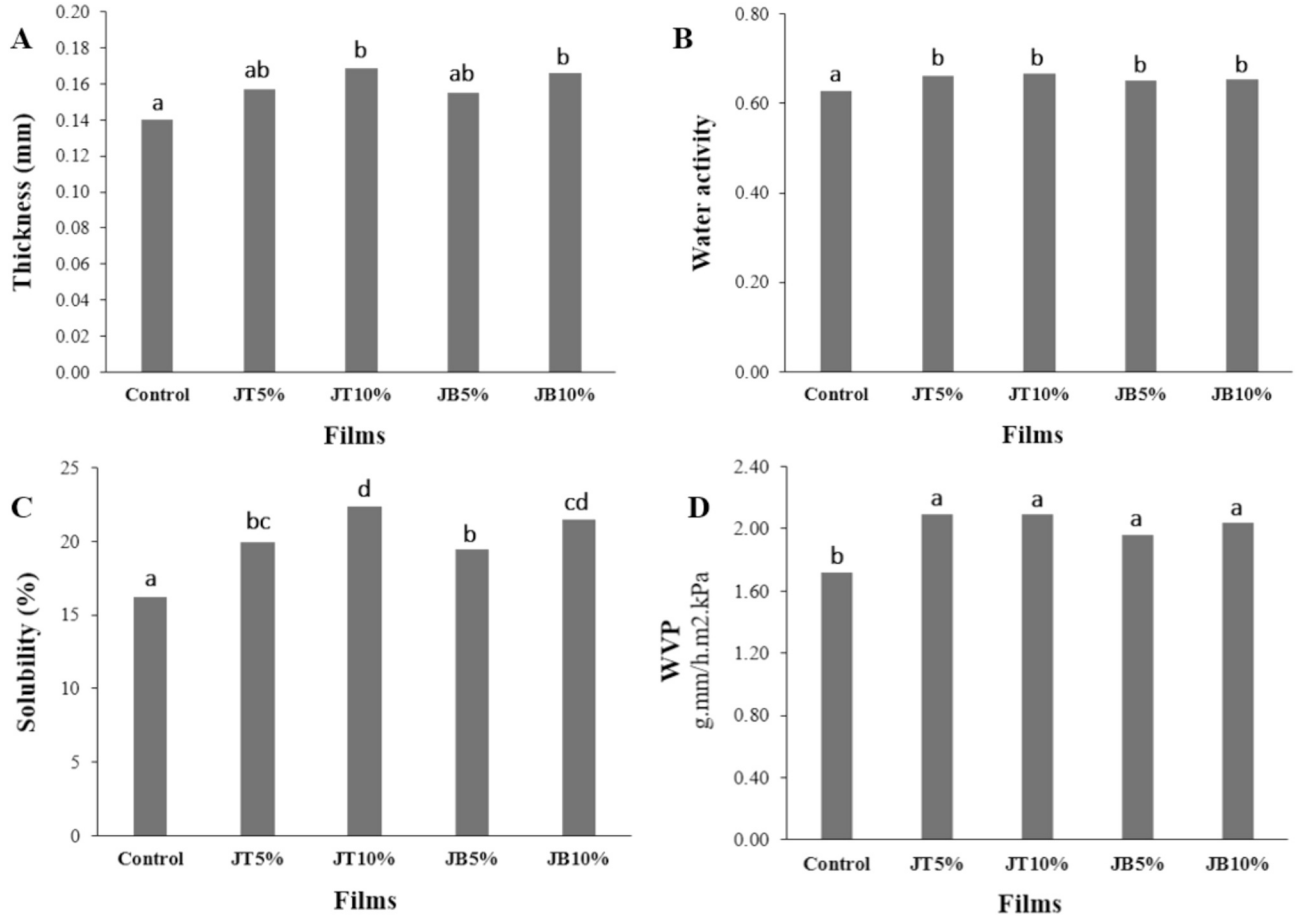


**Fig. 3.** Results of thickness, water activity, water solubility and water vapor permeability (WVP) of the developed films. Means followed by the same letters in the same column do not differ significantly at 5% by Tukey's test.

extracts showed higher total phenolic content and antioxidant capacity, as evidenced by DPPH, ABTS, and FRAP assays (Section 3.3). Phenolic compounds are known to simultaneously absorb UV–visible radiation and act as free radical scavengers; therefore, the higher opacity of these films, combined with their antioxidant capacity, may contribute to the protection of oxidizable foods by limiting photoinduced oxidation.

### *3.4. Thickness, water activity, moisture content, water solubility, and water vapor permeability*

The thickness of the developed films ranged from 0.14 (Control) to 0.17 mm (JT10%). The thickness of the films with 10% jabuticaba and jambolan extracts was similar ($p > 0.05$). The thicknesses that did not differ ($p > 0.05$) from the control film were those with 5% extract added (Fig. 3A). These findings suggest that higher extract concentrations may promote the formation of more complex matrices through anthocyanin–starch interactions, resulting in thicker materials [52].

The increase in thickness with higher extract concentrations can be attributed to the presence of hydrophobic and hydrophilic compounds in jabuticaba or jambolan, which influence the formation of a thicker and more porous structure [8]. The findings of the present study are consistent with those reported by Bodana et al. [8], who evaluated the effects of varying concentrations of pomegranate peel extracts on jackfruit seed starch–based films and observed greater thickness with increasing extract concentration. Similarly, Filipini, Romani, and Martins [14] also reported that incorporating higher concentrations of jambolan extracts into methylcellulose films resulted in increased thickness.

According to Nogueira et al. [53], films developed with fruit-derived fractions should present low water activity to ensure material stability against enzymatic reactions at room temperature, microbial proliferation, and hydrolytic activity. The water activity of all films remained below 0.68 (Fig. 3B). Lower water activity was identified in the control film, which differed from the others ($p < 0.05$), but there was no difference in the extract-added films ($p > 0.05$) (Fig. 3B). No significant differences were observed between the films containing jabuticaba or jambolan extracts, nor between the concentrations tested (5% or 10%) ($p > 0.05$).

The moisture content of the films was significantly higher ($p < 0.05$) after the incorporation of 10% extracts, whether from jabuticaba or jambolan, compared to the control films and those containing 5% extracts. This increase may be attributed to the greater hydrophilicity of the films resulting from higher extract content. The average moisture content ranged from 9.37 ± 0.92% to 13.22 ± 2.82%, which is comparable to the values reported by Nogueira et al. [54] for arrowroot-starch-based films incorporated with blackberry pulp (11.30–12.53%).

Additionally, it is important to emphasize that the moisture content of the films may also affect the microstructure of the starch network, since water molecules can be retained within the micropores present in the matrix [23], consequently influencing water solubility, water vapor permeability, mechanical properties such as flexibility, and the micrographs, as discussed in the following sections.

Solubility values ranged from 16.21% (JT10%) to 22.36% (JB10%) films exhibiting the highest values, followed films containing 5% extracts, which also differed from the control formulation (Fig. 3C). The JT5% and JB10% samples showed similar solubility values ($p > 0.05$).

According to Rodrigues et al. [55], one of the most important factors in developing materials for potential food applications is their resistance

and structural integrity upon contact with water, which can influence solubility. In the present study, the higher solubility observed in films containing greater extract concentrations may be attributed to the presence of anthocyanins, compounds with a strong affinity for water molecules [56]. This increases the hydrophilic regions of the films, enhancing their interaction with water and facilitating solubilization, even after starch–anthocyanin interactions [23,57].

The increase in the hydrophilic fraction of sorghum starch–based films, promoted by the addition of the extract, enhanced the material's affinity for water, thereby facilitating its solubilization. According to Bodini et al. [58], these results indicate that the extract may have acted as a plasticizing agent in the films, leading to a reduction in matrix cohesion and, consequently, to increased polymer chain mobility, which contributed to water uptake and dissolution. This behavior may be associated with the presence of sugars and soluble fibers in the extracts, as reported by Nogueira et al. [23].

It is important to note that there is no ideal value for water solubility, as this property depends on the intended application of the developed material [27]. Low water solubility is desirable when packaging high-moisture foods, whereas high solubility is required for materials used as encapsulants, edible coatings consumed with the packaged product, or even when consumed alone [23,59].

The water vapor permeability (WVP) values of the films ranging from 1.77 (control) to 2.09 g.mm/h.m$^2$.kPa (JT5%). The control sample exhibited the lowest values, whereas JT5%, JT10%, and JB10% films showed the highest results ($p < 0.05$).

Starch-based films typically exhibit low barrier properties, resulting in higher WVP due to the presence of hydrophilic pores that facilitate vapor passage [60]. In addition to the polymer's hydrophilicity, film thickness also affects WVP [61]. In the present study, thicker films (JT10% and JB10%) exhibited higher permeability values.

A potential strategy to improve this property in future study would be the incorporation of hydrophobic components, such as essential oils, into the film formulations to reduce the availability of free molecules capable of binding with water. Another alternative involves the incorporation of nanofibers extracted from jabuticaba peel, since, as reported by Fronza et al. [30], these materials have emerged as promising reinforcement agents for packaging applications, leading to improvements in water vapor permeability and other essential properties, such as moisture control, mechanical strength, and elasticity of packaging materials.

Nevertheless, the films developed in this study exhibited lower WVP compared to materials reported by More, Pegu, and Arya [62], who characterized taro starch films with pomegranate peel extracts, and by Leon-Bejarano et al. [63], who developed potato starch–based films containing hazelnut by-product extracts, suggesting their potential for future applications.

**Table 4**
Mechanical properties of films made with sorghum starch added with jabuticaba or jambolan extracts in different concentrations.

| Films* | TS (MPa) | ε (%) | PD (%) | PR (MPa) |
|---|---|---|---|---|
| Control | 2.66 ± 0.14$^{a}$ | 21.42 ± 3.60$^{b}$ | 16.50 ± 0.05$^{a}$ | 3.97 ± 0.75$^{a}$ |
| JT5% | 1.79 ± 0.34$^{bc}$ | 22.66 ± 5.74$^{ab}$ | 17.01 ± 0.41$^{a}$ | 3.70 ± 1.08$^{a}$ |
| JT10% | 2.09 ± 0.13$^{bc}$ | 26.86 ± 2.06$^{a}$ | 16.85 ± 0.39$^{a}$ | 2.79 ± 0.79$^{a}$ |
| JB5% | 2.14 ± 0.26$^{b}$ | 21.86 ± 4.66$^{ab}$ | 16.99 ± 0.42$^{a}$ | 3.00 ± 0.61$^{a}$ |
| JB10% | 1.65 ± 0.40$^{c}$ | 23.93 ± 2.79$^{ab}$ | 16.70 ± 0.33$^{a}$ | 2.88 ± 0.29$^{a}$ |

- Means followed by the same letters in the same column do not differ significantly at 5% by Tukey's test. TS: tensile strength. ε: elongation at break. PR: puncture resistance and PD: puncture deformation.

* Control (film without addition of extract), JT5% and JT10% (Films with addition of 5% and 10% of jabuticaba extract respectively), JB5% and JB10% (Films with addition of 5% and 10% of jambolan extract respectively).

### 3.5. Mechanical properties

The effect of incorporating jabuticaba and jambolan peel extracts into sorghum starch films was evaluated in terms of mechanical properties, including tensile strength (TS), elongation at break (ε), puncture resistance (PR), and puncture deformation (PD), as shown in Table 4.

The tensile strength (TS) of all films decreased with the addition of jabuticaba or jambolan extracts, regardless of the concentration, compared to the control sample ($p < 0.05$) (Table 4). The lowest TS was recorded at JB10% (1.65 MPa), followed by JT5% (1.79 MPa). According to Prietto et al. [64], films prepared solely with starch form an organized three-dimensional network through intermolecular interactions after gelatinization. However, the presence of anthocyanins may weaken these intermolecular interactions, thereby reducing the TS of the films.

In turn, the increase in ε observed in JT10% (26.86%) film resulted in a more flexible material compared to films prepared without extract (Table 4). According to Cunha et al. [65], the added extract may act as a plasticizer due to its strong interaction with the starch used as the matrix polymer, leading to higher ε values (%). The polar components present in the extract can form molecular interactions with hydroxyl groups, replacing those previously formed solely by starch molecules.

Additionally, Martelli et al. [66] highlights that sugars, fibers, and proteins present in fruit peels can act as plasticizers, contributing to films that are less resistant but more flexible. Flexibility is a desirable property when applying the developed material as a food coating, since the coating must not be rigid in order to fully cover the product [15]. Regarding the effect of adding different extract concentrations to films, Silva et al. [67] observed that increasing jabuticaba extract levels (15, 30, 45, and 60%) in cassava starch–based films also influenced the mechanical properties of the formulations, as evidenced by higher elongation percentages.

The addition of jabuticaba or jambolan peel extracts did not affect the puncture-related mechanical properties ($p > 0.05$). A similar trend was reported for potato starch films containing hazelnut peel extracts by Leon-Bejarano et al. [63]. Thus, the extracts may have acted as secondary plasticizing agents, increasing the mobility of starch chains (resulting in higher elongation at break) without compromising the local cohesion of the matrix, which governs puncture behavior.

### 3.6. SEM films

The SEM micrographs revealed the surface morphology of the developed films. No cracks were observed; however, the film produced solely with sorghum starch exhibited a more homogeneous and smoother surface compared to the others (Fig. 4). The incorporation of extracts led to the formation of surface roughness, which increased proportionally with the extract concentration. Consequently, films containing 10% (JT and JB) extract displayed a visually denser surface morphology.

This feature may be related to the thickness, opacity, and mechanical properties of the films. According to Silva et al. [15], the presence of surface roughness suggests that the incorporation of extracts promoted strong interactions among the polymeric constituents. The same authors also reported that the increased opacity observed in films is directly associated with surface roughness. In this context, the irregular surfaces of films containing extracts may account for the reduction in their mechanical properties compared to the control films [68].

Similar morphological alterations have also been reported in other types of materials upon the incorporation of plant extracts. Silva et al. [22] observed such modifications in yam starch films containing aroeira leaf extracts, whereas Qin et al. [69] reported analogous results in cassava starch films incorporated with *Lycium ruthenicum* extract.

Jabuticaba and jambolan peel extracts are chemically complex matrices containing both hydrophilic and less polar compounds. Hydrophilic constituents include anthocyanins, phenolic acids, sugars, and

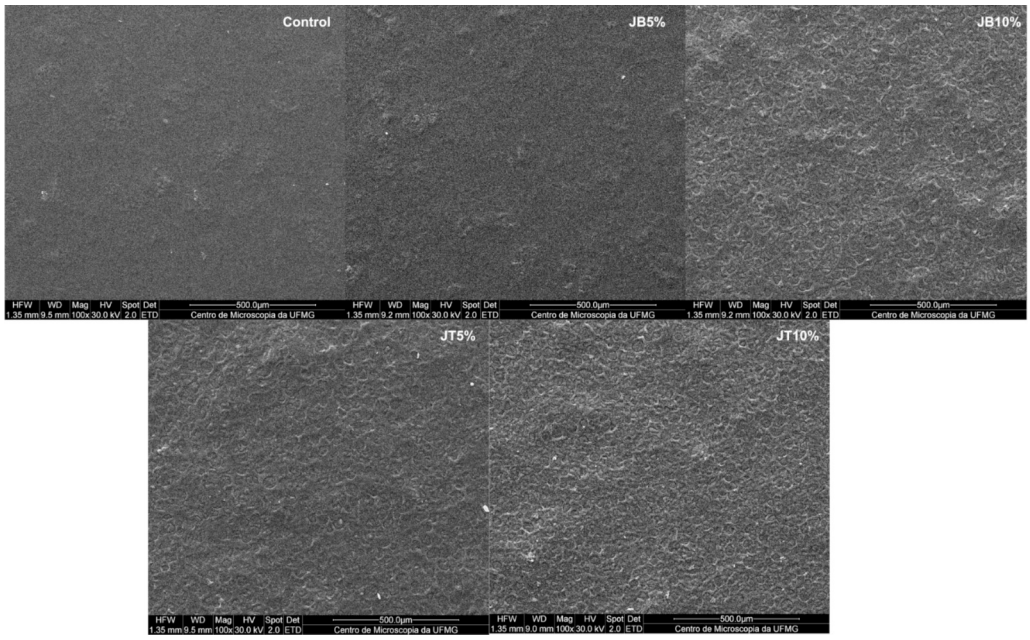


**Fig. 4.** Micrographs of the surfaces section of the sorghum starch films without jaboticaba and jambolan peels extracts (control), with 5% and 10% jambolan peel extract (JB5% and JB10%) and with 5 and 10% jaboticaba peel extract (JT5% and JT10%).

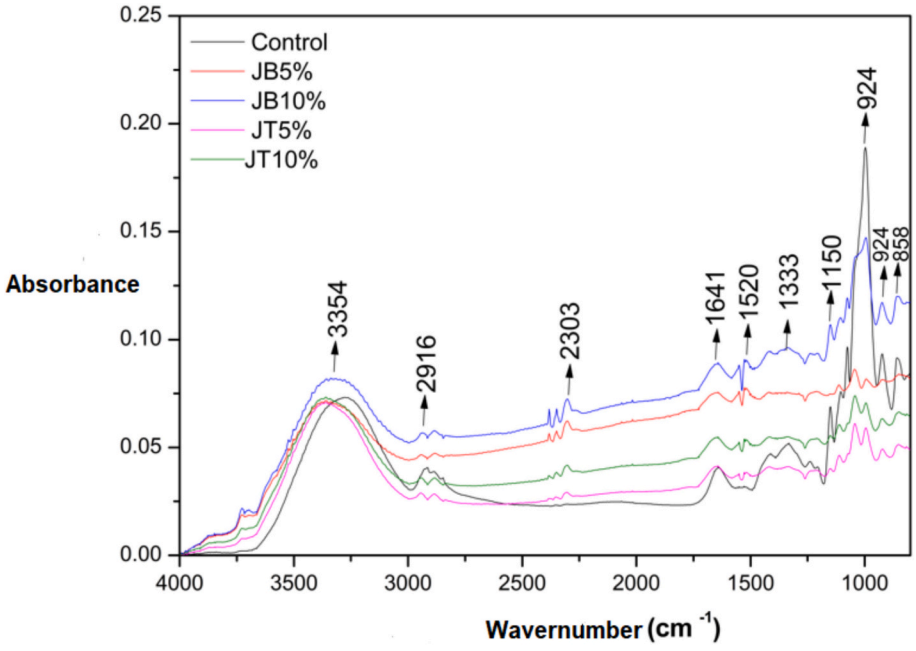


**Fig. 5.** Fourier transform infrared spectra of the developed films without jaboticaba and jambolan peels extracts (control), with 5% and 10% jambolan peel extract (JB5% and JB10%) and with 5 and 10% jaboticaba peel extract (JT5% and JT10%).

organic acids, whereas less polar or hydrophobic components such as flavonoid aglycones, condensed tannins, lipophilic phenolics, and residual waxy compounds from the fruit peels may also be present [12,13]. At the microscale, the coexistence of compounds with different polarities may affect intermolecular interactions within the starch matrix, leading to heterogeneous molecular organization. These effects may contribute to the formation of thicker film structures as extract concentration increases, which is consistent with the thickness values observed and previously reported in this study.

### 3.7. FTIR and thermogravimetric analysis (TGA)

Infrared spectra were obtained to evaluate the interactions between the film structures, as shown in Fig. 5. All films exhibited characteristic bands of thermoplastic materials based on starch and glycerol, including OH bond stretching (around 3300 $cm^{-1}$) and CH bonds (at 2916 $cm^{-1}$) [70].

The addition of extract, either of jabuticaba or jambolan, resulted in shift and broadening of the band associated with hydroxyl groups (~3350 $cm^{-1}$). In other words, as the extract concentration increased, the intensity of the –OH stretching peak also increased and shifted to a

higher wavenumber due to hydrogen bonding between the molecules present in the extract and the starch matrix [71]. This effect may be associated with the increased moisture content of the material as the extracts were added, as well as with changes in the mechanical properties of the films, such as flexibility and TS, due to hydrogen bond cross-linking [72].

The films JT10%, JT5%, JB10%, and JB5% containing anthocyanins showed an increase in peak absorbance at 1641 $cm^{-1}$, suggesting that the anthocyanins were immobilized in the starch–glycerol matrix, resulting from the stretching vibration of the C—C aromatic ring [17]. Meanwhile, the peak at 1520 $cm^{-1}$, observed only in films containing extracts, corresponds to the stretching vibration of C═O bands and the bending vibration of C–O–H bands, which is related to the presence of polyphenolic compounds [73], compounds found in the aqueous extracts of jabuticaba and jambolan peels [12].

Rodrigues [74] reported that the peaks between 2400 and 2300 $cm^{-1}$ are indicative of interactions between starch and proteins, which can occur in plant-based materials. Additionally, other peaks observed in the FTIR spectrum are those at 1150 $cm^{-1}$ and 921 $cm^{-1}$, representing the stretching of the C—O bond in granular starch. These same peaks were also observed by Chaves-Marquez et al. [17], who developed intelligent and active potato-starch films incorporated with purple corn cob extracts.

The region from 1200 to 800 $cm^{-1}$ is considered the fingerprint region for polysaccharides, showing bands related to the C–O–C and C–O–H stretching vibrations of glycosidic bonds [50]. From the spectra obtained, it can be observed that the addition of extract did not alter the conformation, only the intensity of these bands.

Thermogravimetric analysis (TGA) was performed to evaluate the degradation and thermal stability of sorghum starch–glycerol films incorporated with jabuticaba or jambolan extracts (Fig. 6).

Three stages of degradation were observed. In the first stage (~120 °C), the downward trend of the TGA curve was smooth, and the observed mass loss (≈12.0%) can be attributed to the evaporation of water from the film through dehydration, as well as the volatilization of other small molecules in the polymer matrix [50]. The second stage (from 120 °C to 230 °C) corresponds to the decomposition of low-molecular-weight organic materials, such as glycerol and phenolic compounds (present in the samples containing extracts). According to Rodrigues et al. [75], in this temperature range, starch depolymerization and internal reorganization may occur, causing the glass transition phenomenon, in which the polymeric matrix modifies its characteristics.

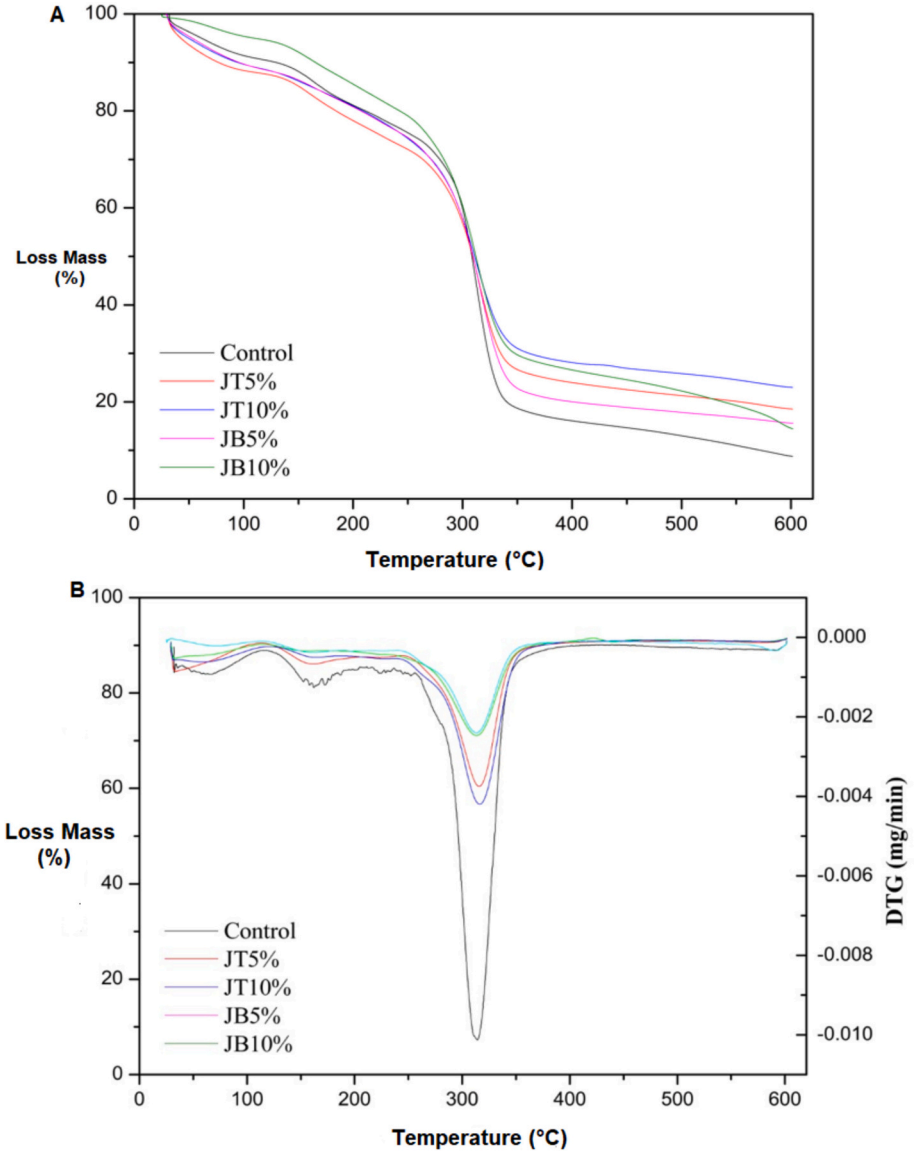


**Fig. 6.** TGA (A) and DTG (B) curves of films made with sorghum starch added with extracts of freeze-dried jabuticaba (JT) and jambolan (JB) peels.

Finally, the steepest mass loss of all analyzed materials occurred in the temperature range of approximately 230 °C to 380 °C, with a mass loss of ≈ 65% and a DTG peak around 320 °C. This event is related to the decomposition of the compounds comprising sorghum starch, due to the disruption of adjacent hydroxyl and glycosidic bonds in the polymer matrix, accompanied by the oxidation and decomposition of organic matter [50]. Additionally, minor losses can occur above 450 °C, attributed to the degradation of by-products formed during the analysis [76]. No further events were observed above 595 °C, indicating the predominance of inorganic components [30].

In general, all films exhibited good thermal stability, being able to withstand high temperatures, which indicates the formation of an organized and stable matrix. Thus, the addition of extract did not affect the thermal behavior of the developed materials.

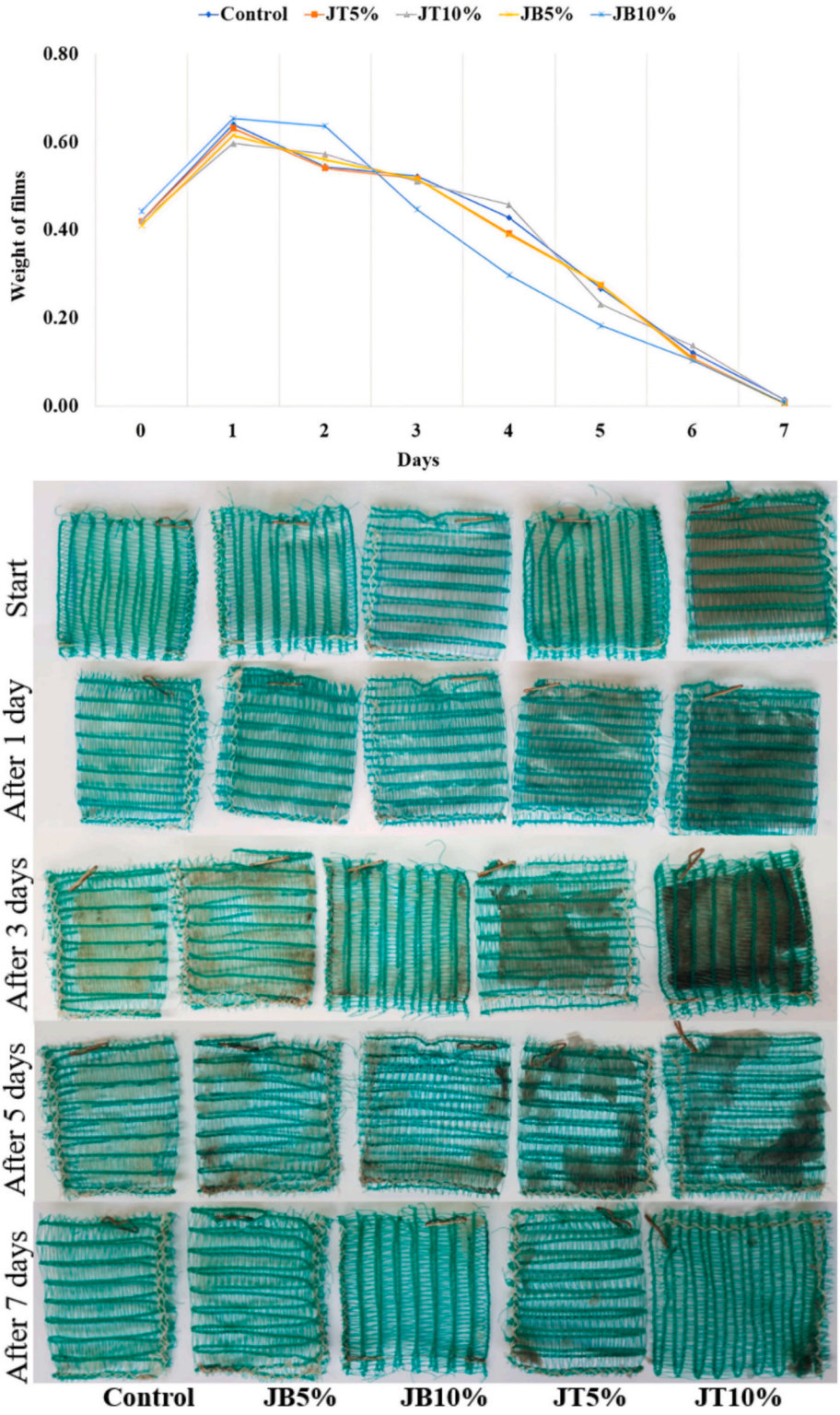


**Fig. 7.** Degradation graph and visual assessments of soil biodegradability tests.

### 3.8. Biodegradation

In the evaluation, the biodegradation of the films in soil, carried out quantitatively, through degradation/weight loss graph, and qualitatively, through visual observations, a reduction in the film areas was observed over the evaluation period (0, 1, 3, 5 and 7 days) (Fig. 7). All samples were completely degraded within this period, confirming their potential for application in the development of packaging for food products and/or other sectors.

During the seven days of soil burial of the films, all samples exhibited structural breaks and changes in their color, indicating the onset of degradation from the second day onward (Fig. 7). The incorporation of jabuticaba and jambolan extracts did not influence the biodegradation properties of the developed films.

The water added to the system diffuses through the soil and promotes the swelling of the films under analysis, increasing their initial weight. This process also leads to cracks and fragmentation of the films over time [77]. Additionally, due to the hydrophilic nature of starch-based films, they rapidly absorb moisture, resulting in the breakdown of hydrogen bonds and intermolecular interactions formed between the film components. This facilitates the action of soil microorganisms and naturally occurring enzymes [14,78].

Some studies in literature report the degradation of films made from different starch sources over time [78–80]. For example, Jaramillo et al. [79] reported the complete biodegradation of films made from cassava starch within 12 days. Jiang et al. [80] observed that active films based on longan seed starch incorporated with anthocyanin-rich extracts were fully degraded within 30 days. Similarly, Li et al. [78], when studying films composed of pea starch nanofibers, reported significant mass loss from the third day of soil burial.

According to Filipini, Romani, and Martins [14], to be considered biodegradable, 90–95% of a material's structure must be degraded by biological action within six months. Therefore, all films produced in the present study can be considered biodegradable and represent an important alternative for reducing costs associated with the processing and recycling of petrochemical-based plastic materials.

From an application standpoint, the characteristics exhibited by the sorghum starch–based films incorporated with fruit extracts, particularly their high moisture affinity, water solubility, and water vapor permeability, indicate promising potential for use in food systems where direct contact with high-moisture matrices is not required. In this context, these materials may be especially suitable for applications involving foods with low water activity, such as bakery products, dry snacks, or as inner packaging components for dehydrated products, including tea sachets and herbal infusions. In addition to their barrier and mechanical functionality, the incorporation of fruit extracts confers added value by introducing bioactive compounds, which may contribute antioxidant protection and enhance the functional profile of the packaged product.

Therefore, application trials in actual food matrices under realistic storage conditions represent a necessary next step to validate the practical performance of these films and are currently being addressed in ongoing studies.

## 4. Conclusion

The sorghum starch-glycerol films combined with different concentrations of jabuticaba or jambolan peels extracts exhibited good appearance and handling flexibility, without fractures or visible blisters. The incorporation of 10% extract imparted a reddish/purplish color to the films, a characteristic that may be attractive for future applications. The addition of extracts increased the anthocyanin content of the formulations and led to higher values of thickness, water activity, moisture, water solubility, and water vapor permeability. All developed films demonstrated good thermal stability, the presence of functional groups, and complete degradation within seven days.

However, the inherent limitations of sorghum starch-based films, particularly their lower mechanical strength and barrier performance affected by moisture, solubility, and water vapor permeability, remain challenges to be addressed. Improvement of these properties may be investigated in future studies through the incorporation of hydrophobic materials, such as essential oils, to reduce water permeability, or nanofibers, aimed at enhancing mechanical strength and reinforcing the film structure. Despite these limitations, the films demonstrate potential for application in food packaging, including use as quality indicators, which should be evaluated through specific tests in food matrices.

## CRediT authorship contribution statement

**Vinícius Tadeu da Veiga Correia:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Nayana Hayss Araújo Silva:** Formal analysis. **Pâmella Fronza:** Writing – original draft, Formal analysis. **Ana Luiza Santos Vieira:** Formal analysis. **Ana Luiza Mendes Nunes da Silva:** Methodology, Formal analysis. **Isabella Maciel Costa:** Methodology, Formal analysis. **Taynan Jonatha Neves Costa:** Formal analysis. **Bruna Maria Salotti de Souza:** Writing – review & editing, Methodology, Formal analysis. **Valéria Aparecida Vieira Queiroz:** Writing – review & editing, Resources, Formal analysis. **Washington Azevedo da Silva:** Formal analysis. **Julio Onesio-Ferreira Melo:** Writing – review & editing, Supervision, Resources, Conceptualization. **Camila Argenta Fante:** Writing – review & editing, Supervision, Resources, Conceptualization.

## Declaration of competing interest

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

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## Data availability

No data was used for the research described in the article.

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