Research Square

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Development of sodium alginate-based soluble active sachets containing jatobá (Hymenaea sp.) extract for whole milk powder preservation

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Abstract

Whole milk powder requires effective packaging to maintain its quality and safety, while biodegradable materials offer a potential alternative to conventional packaging. This study aimed to develop sodium alginate films containing jatobá (*Hymenaea* sp.) pulp extract and evaluate their potential application as soluble active sachets for whole milk powder. Films containing 0, 10, 20, and 30% (v/v) extract were characterized for phenolic content, antioxidant activity, physical, mechanical, barrier, and optical properties. Selected formulations were used to prepare sachets, which were evaluated during 50 days of storage at 30°C. The incorporation of jatobá extract increased the total phenolic content and antioxidant activity of the films. At the highest extract concentration, tensile strength increased 2.2-fold and elongation at break increased by 90% compared with the control. Extract concentrations of 20 and 30% reduced water solubility and increased water vapor permeability, whereas the 10% formulation maintained barrier properties comparable to those of the control and was selected for sachet production. During storage, milk powder packaged in extract-containing sachets exhibited lower thiobarbituric acid reactive substances (TBARS) and titratable acidity than samples stored in the other packaging systems. The sachets dissolved at both 37 and 50°C, with less residual material observed at the higher temperature. These findings highlight the potential of jatobá extract-containing alginate sachets as a soluble active packaging alternative for powdered foods.

1 Introduction

Whole milk powder is an essential component of many people's diets worldwide, and packaging plays a crucial role in preserving its quality and safety [32, 37]. Due to its lipid content, whole milk powder is susceptible to quality deterioration during storage, particularly through lipid oxidation and changes associated with moisture and oxygen exposure [26, 37]. Therefore, packaging must provide adequate protection against environmental factors that may compromise product quality and stability [13, 26, 37, 53]. In this context, the development of biobased and biodegradable packaging materials has gained attention as an alternative to conventional petroleum-based packaging, particularly when new materials can combine product protection with reduced solid-waste generation.

Among the alternatives investigated for powdered foods, soluble films and sachets are particularly interesting because the packaging can be dissolved together with the product during preparation, potentially reducing the need for disposal after use. Biopolymer-based edible films containing sodium alginate, agar, or inulin have been proposed for powdered foods and beverages requiring dissolution or reconstitution [33]. More recently, water-soluble edible films have been developed for instant coffee and peptide powder packaging, demonstrating the feasibility of converting soluble films into sachet-type systems for powdered products [49, 50]. Whey protein isolate-based films have also been investigated for fast-dissolving and heat-sealable pouches containing premeasured dry foods or ingredients [23].

Despite these advances, the development of soluble packaging for powdered products presents a technological challenge: the material must dissolve during preparation while maintaining sufficient

physical integrity during handling and storage. This requires a balance between rapid dissolution, mechanical properties, and barrier performance [12]. Highly hydrophilic biopolymer matrices may favor water solubility but can also be susceptible to moisture uptake, making the simultaneous evaluation of solubility, mechanical properties, and water vapor permeability (WVP) important when selecting materials for soluble sachets [23, 33, 50]. Among the biopolymers investigated for edible and biodegradable films, sodium alginate is a promising film-forming material for incorporating bioactive compounds. However, its hydrophilic character can influence moisture-related barrier properties, highlighting the need to characterize its performance according to the intended packaging application [25].

The incorporation of bioactive compounds into such matrices provides an additional technological function. Active packaging systems containing antioxidant compounds can be designed to provide protection against oxidative deterioration of packaged foods, complementing the passive barrier properties of the packaging material.This approach is particularly relevant to whole milk powder because lipid oxidation is one of the important quality changes occurring during storage [21], for example, investigated an antioxidant multilayer package containing α-tocopherol for whole milk powder and reported a reduction in the oxidation rate of the product. These findings support the potential of antioxidant packaging as a complementary strategy for protecting milk powder against oxidative deterioration.

Natural compounds, particularly plant extracts rich in phenolic compounds, have been investigated as active components in biopolymer-based films. In particular, several studies have demonstrated that the incorporation of plant extracts into alginate matrices can modify antioxidant, mechanical, barrier, and optical properties of the resulting films [25, 47]. *Hymenaea sp*, a fruit locally known as jatobá, is characterized by its starchy texture, mild sweetness, and distinctive flavor. It is appreciated both in its natural form and as an ingredient in various culinary preparations [8, 39]. However, despite being underexplored technologically, jatobá pulp has remarkable antioxidant potential. Studies have revealed that jatobá pulp has a high content of phenolic compounds and tannins, which are directly associated with its antioxidant activity[2, 3, 8].

Several studies have investigated biopolymer-based soluble sachets for food packaging, including chitosan sachets containing *Santalum* essential oil to delay butter oxidation [18], pectin sachets containing olive leaf extract for powdered chicken broth [38], and a blend of collagen and methylcellulose for powdered coffee, salt, and biscuits [44]. These studies demonstrate that soluble sachets can be developed using different biopolymer matrices and active compounds. However, the integration of a sodium alginate matrix and jatobá pulp extract into a soluble active sachet intended for whole milk powder remains insufficiently explored. Therefore, the present study aimed to develop and characterize active films of sodium alginate incorporated with extracts from jatobá pulp and use them as a basis for the production of soluble sachets, that were applied as packaging for whole milk powder.

2 Material and methods

2.1 Material

Ripe jatobá fruits (*Hymenaea sp.*) were collected in the region of Campo Grande, MS, Brazil. The sodium alginate (CAS 9005-38-3) and glycerol (CAS 56-81-5) used in the preparation of films were purchased from Êxodo Científica (Sumaré, Brazil). Milk powder (Elegê) was purchased in the local market (Rio Grande, RS, Brazil). All other solvents and chemicals used were of analytical grade.

2.2 Preparation and extraction of bioactive compounds from jatobá pulp

The jatobá fruits were washed and sanitized, then pulp and shell were separated, and dried for 6 h at 40°C in a forced air circulation oven (Fanem 520-C, São Paulo, Brazil). Later, the dry pulp was milled in a blender (Arno 700W-PL, São Paulo, Brazil) and its granulometry was standardized using a sieve with mesh 42 (0.380 mm).

The hydroalcoholic extract (30% ethanol) of pulp was prepared using with 5 g of pulp powder. Solvents and pulp were homogenized on an orbital shaker (Tecnal TE-420, Piracicaba, Brazil) at 25°C for 1 h at 150 rpm. Then, the solution was centrifuged at 8709 ×*g* for 10 min and the supernatant was filtered through a filter paper under vacuum at room temperature (25 ± 1 ºC). The pulp extract (PE) was kept at 5 ± 1 ºC in amber glass and used in the films preparation on the same day.

2.3 Production of active films

The films were obtained through the casting technique by homogenization of 1.5 g of sodium alginate in 100 mL of distilled water under mechanical stirring (Fisatom, 712, Brazil) at 1000 rpm for 30 min at 70 ºC. Then, a plasticizer was added (0.6 g of glycerol. 100 mL^{-1}) and the mixture was stirred again for 15 min. The solution was cooled at 40 ºC and 10, 20, or 30% (v/v) of PE were added and homogenized for more 15 min under mechanical stirring (films added of pulp were named JEF). A control film (CF) was developed without the addition of the extract (0%). For film formation, 15 g of the filmogenic solution were poured into Petri dishes (Ø = 9 cm) and dried in a forced air circulation oven for 24 h at 40°C. The films were conditioned in desiccators with a saturated solution of sodium bromide at 25°C and 55% of relative humidity (RH) for 24 h before analysis.

2.4 Films characterization

All film formulations (CF, JEF10, JEF20, and JEF30) were characterized before the selection of the formulations for sachet production. The characterization included phenolic content and antioxidant activity, physical, mechanical and water vapor barrier properties, and optical properties. The results of these analyses were considered together to identify the formulations most suitable for the subsequent sachet application.

2.4.1 Phenolic compounds and antioxidant activity of films

The total phenolic compounds (TPC) and antioxidant activity of films were evaluated using aqueous extracts. The preparation of the sample was performed using Santos et al [41]. Briefly, 150 mg of films were immersed in 10 mL of distilled water and subjected to extraction in a shaker at 150 rpm for 3 h at 25 ºC. Subsequently, the solution was centrifuged at 8709 ×*g* for 10 min and filtered on filter paper, obtaining the aqueous extracts of the films. TPC quantification was performed by the Folin-reagent method described by Oliveira Filho et al. [31]. Antioxidant activity of the films was determined by $ABTS^{\cdot+}$ and DPPH using the methods described by Filipini et al. [15]. The TPC and antioxidant activity were expressed as milligrams of gallic acid equivalents per gram of film (mg GAE/g film).

2.4.2 Physical, mechanical, and barrier properties of films

Film thickness was measured in triplicate, with a micrometer (Insize IP54, precision 0.001 mm) at ten random positions for each film sample. The mechanical properties, tensile strength (TS) and elongation at break (EB), of films were evaluated according to the ASTM D-882-02 method (ASTM, 2002).

The film's solubility in water (SW) was obtained according to Alves-Silva et al. [4]. Water vapor permeability (WVP) was gravimetrically determined according to the method described by ASTM Standard Method E96-00 (ASTM, 2000).

2.4.3 Optical properties of films

Color and opacity were measured using a colorimeter (Minolta CR 400, Tokyo, Japan) with the CIELab color space. The colorimeter was calibrated with an Illuminant D65. The L_0 parameters (luminosity/brightness) and the total color difference (ΔE) were analyzed. ΔE was calculated using the values of a* and b* parameters, according to Eq. (1), where L* = 97.39, a* = 0.14, and b* = 1.94 were the default color parameters and L_0, a_0, and b_0 were the films color parameters.

$$\Delta E = [(L^*-L_0)^2 + (a^*-a_0)^2 + (b^*-b_0)^2]^{1/2} \quad (1)$$

Using the same colorimeter previously mentioned, the opacity (%) of each film was determined by the ratio of the black (Y_b) and white (Y_w) standards.

2.5 Evaluation of the effect of a soluble film as a sachet for whole milk power

2.5.1 Sachets preparations

Following the characterization described in Section 2.4, two film formulations, CF and JEF10, were selected for the sachet application. The selection was based on the combined results for water solubility and water vapor permeability, considering the need for the material to dissolve during product preparation while providing suitable properties for handling and storage. The selected films were folded into rectangular sachets measuring 5 × 15 cm and thermally sealed. Each sachet was then filled with 13 g of whole milk powder and sealed before storage.

Three packaging conditions were evaluated: sachets prepared from the jatobá-extract-containing film (JES), sachets prepared from the control film without extract (CS), and commercial polypropylene packaging (SS), under the same conditions (Fig. 1). The samples were stored at 30°C for 50 days. Whole milk powder quality was evaluated at 7-day intervals

2.5.2 Evaluation of whole milk powder quality during storage

2.5.2.1 Lipid oxidation (TBARS), titratable acidity, pH, moisture and color

Thiobarbituric acid reactive substances (TBARS) were measured by the method of King (1962) and the results expressed as absorbance at 532 mm in a spectrophotometer UV-VIS (Kasuaki IL592, Brazil). Titratable acidity and pH were carried out in triplicate, following the methodologies of Instituto Adolfo Lutz [22]. The titratable acidity of WMP was expressed as lactic acid. For analysis, 1 g of sample was diluted in 7 g of water and titration was performed using a sodium hydroxide solution (0.1 N) using phenolphthalein as an indicator.

Moisture content was determined according to the methods described in the Association of Official Analytical Standards (AOAC), by oven drying method at 105°C for 24 h (Method 950.46) [6].

The color of whole milk powder was measured using a colorimeter (Minolta CR 400, Tokyo, Japan) with the CIELab color space, analyzing the parameter b* (yellowness/blueness). For analysis, 2 g of sample were placed in a clear petri dish, and color measurement in triplicate.

2.5.2.2 Reconstitution properties of whole milk powder

The dispersibility and insolubility index (ISI) of whole milk poder were evaluated following the methods described by the Instituto Adolfo Lutz [22]. The milk powder was reconstituted following the preparation instructions provided on the product label (1 g of milk powder to 7 g of water). The required amount of water and milk powder was mixed manually using a spoon for 30 s.

The solubilization behavior of the sachets containing whole milk powder was evaluated separately. The sachets were immersed in distilled water at 37 and 50°C. After the test, the resulting aqueous dispersion was filtered, and the presence of solid residues in the filtrate was visually assessed and documented photographically.

2.6 Statistical analysis

All analyses were performed at least in triplicate and standard deviations were reported. Analysis of variance (ANOVA) was used, and Tukey's test was applied to determine the differences between the samples at 95% confidence using the Statistic 5.0 (Stasoft, USA) software.

3 Results and discussion

3.1 Films characterization

3.1.1 Phenolic compound content and antioxidant activity

Jatobá pulp is recognized for containing diverse bioactive compounds [3], including phenolic compounds, flavonoids, tannins, fatty and phenolic acids, procyanidins and coumarins [8, 39]. Generally, phenolic compounds are renowned for their antioxidant properties and their capacity to form chemical bonds, which can impact the physical properties of films [41]. Consequently, the analysis of total phenolic compounds (TPC) and the antioxidant activity of the films developed in the present study assumes a crucial role in assessing the antioxidant potential resulting from the incorporation of jatobá pulp extract. Results of TPC and antioxidant activity are presented in Fig. 2.

The Films demonstrated a significant increase ($p < 0.05$) in total phenolic compound content with the addition of extracts to alginate films (Fig. 2A). The control film exhibited a TPC of 0.5 mg GAE g^{-1} film (Fig. 2A), whereas the extract-containing films presented values ranging from 7.8 to 17.9 mg GAE g^{-1} film, with TPC increasing as the extract concentration increased. This concentration-dependent response indicates that the incorporation of jatobá pulp extract increased the amount of phenolic compounds measured in the film formulations. The antioxidant activity also increased with extract incorporation in both assays (Fig. 2B). These results are consistent with previous studies reporting increased antioxidant activity in biopolymer films containing plant extracts, with the response associated with the concentration of the incorporated extract [14, 31, 40].

Mean ± standard deviation. Different letters in the same image indicate significant differences ($p < 0.05$) by Tukey's test

The antioxidant activity values obtained by the DPPH method ranged from 9.8 to 11.4, and the ABTS values ranged from 8.6 to 10 mg GAE g^{-1} film. The two assays provide complementary estimates of radical-scavenging activity, and the increase observed in both supports the antioxidant potential of the extract-containing films. The response may be associated with antioxidant compounds reported in jatobá pulp, including phenolic compounds, flavonoids, and tannins [8, 29].

Considering that oxidation is one of the primary causes of food deterioration, the presence of antioxidant activity in films is advantageous as it can prolong the shelf life of stored products by slowing down or inhibiting the oxidation of the product’s components, thereby maintaining its quality for a longer period [5]. Thus, the antioxidant activity detected in the films supports their potential application in active packaging systems intended for oxidation-sensitive foods. Nevertheless, the extent to which this activity contributes to food preservation must be assessed in the packaged product, since radical-scavenging activity measured in the films alone does not demonstrate an extension of shelf life.

3.1.2 Influence of jatobá pulp extract on the packaging performance of alginate films

All films of the present work showed visually uniform structures without ruptures or flaws. The results of the effects of the addition of extracts on physical, mechanical, and barrier properties of alginate films are presented in Fig. 3. The thickness of biopolymeric films plays an important role in determining their physical and functional properties, directly influencing their performance and applicability. It is intrinsically linked to barrier characteristics, mechanical strength, and durability [40]. The thicknesses ranged between 0.045 mm and 0.056 mm, increasing proportionally with higher concentrations of jatobá extract (Fig. 3A). This pattern is comparable to several documented studies wherein the presence of soluble solids derived from plant extracts increases in material thickness[5, 16, 40]

Mean ± standard deviation. Different letters in the same image indicate significant differences ($p < 0.05$) by Tukey's test

The incorporation of jatobá pulp extract into a sodium alginate matrix resulted in an increase in tensile strength (TS) and elongation at break (EB) (Fig. 3C and 3D, respectively). The maximum extract concentration (30%) led to a 2.2-fold increase in TS and a 90% increase in EB. These results indicate that extract incorporation modified the mechanical behavior of the alginate films, improving both tensile resistance and deformation capacity. Interactions between extract constituents and the polymeric matrix may have contributed to these changes; however, the specific molecular mechanisms responsible for the simultaneous increases in TS and EB cannot be established from the mechanical results alone. Similar results were obtained by Kan et al. [24], who observed that the addition of Chinese hawthorn extract to chitosan-gelatin films increased thickness, tensile strength, and elongation at break.

Although an inverse relationship between TS and EB has been reported for some alginate films containing plant extracts, the present results show that both properties increased with jatobá extract incorporation [5, 40]. This difference suggests that the effect of plant extracts on mechanical performance depends on the composition of the extract and the characteristics of the polymeric matrix rather than following a universal relationship between strength and extensibility.

Among the constituents found in jatobá pulp, it is noteworthy to mention the presence of tannins, which are polyphenolic compounds capable of contributing to the strength and elasticity of materials. Such polyphenols have the ability to form complexes with proteins and polysaccharides [34], exerting influence on the physical properties of films, in addition to their antioxidant properties [28, 40]. In terms of macromolecules, according to Alves-Silva, Romani & Martins [3], jatobá pulp contains 6.5 g of protein and 1.38 g of lipids per 100 g. However, the bulk composition of jatobá pulp alone cannot explain the mechanical behavior of the resulting films. Film properties depend not only on the presence of proteins, lipids, and phenolic compounds but also on their retention during extract preparation, their dispersion

within the polymeric matrix, and their interactions with alginate during film formation [11, 35, 42]. Phenolic compounds may interact with polysaccharides through hydrogen bonding and other molecular interactions, potentially modifying matrix organization and mechanical performance. In this study, the simultaneous increase in tensile strength and elongation at break at higher jatobá extract concentrations indicates that extract incorporation altered the mechanical response of the alginate films. This behavior may reflect the combined effects of extract constituents and their interactions with the polymeric matrix, rather than the contribution of a single component.

The control film exhibited solubility around 70% (Fig. 3E) and permeability around 0.21 g mm kPa^{-1} day^{-1} m^{-2} (Fig. 3F). The addition of 10% of extract to the polymeric matrix did not significantly alter the barrier properties. However, when increased to 20 and 30%, the extracts promoted a reduction in solubility (SW) and an increase in water vapor permeability (WVP). According to Santos et al. [40], phenolic compounds have the ability to cross-link with polysaccharides as alginate, potentially leading to a decrease in film solubility, as cross-links result in a more stable matrix, less susceptible to dissolution.

Additionally, the incorporation of plant extracts into alginate films can lead to an increase in free volume, thereby increasing WVP [7, 40]. The simultaneous decrease in SW and increase in WVP observed at 20 and 30% extract therefore indicates that resistance to dissolution and resistance to water vapor transport did not follow the same trend. This distinction is particularly relevant for soluble sachets intended for powdered foods, the film must retain sufficient integrity during handling and storage while dissolving under the intended conditions of use [50] .

Previous studies on soluble biopolymer-based sachets have investigated formulations combining sodium alginate with other hydrocolloids and plasticizers, as well as alternative protein- and polysaccharide-based systems [23, 33, 51]. These studies provide a broader context for understanding the balance between mechanical performance, water sensitivity, and dissolution in soluble packaging. For the present system, the absence of significant changes in SW and WVP at 10% extract, together with the changes observed at higher concentrations, suggests that extract concentration is an important formulation parameter. Nevertheless, the selection of the most suitable concentration should consider the combined results for mechanical properties, solubility, barrier performance, and the subsequent storage tests of the milk powder, rather than relying on any single property.

3.1.3 Optical characteristics of the films

The evaluation of color parameters in biopolymeric films plays a significant role in characterizing these materials and can provide important insights into their visual appearance and suitability for specific packaging applications [36]. The results of the color and opacity parameters obtained for the sodium alginate films incorporated with different concentrations of JPE are presented in Table 1.

Table 1
Color parameters of control sodium alginate films and incorporated with jatobá pulp extract.

Films	Opacity (%)	L	a*	b*	ΔE
CF	9.86 ± 0.10 ^{c}	95.81 ± 0.45 ^{a}	-0.24 ± 0.01 ^{c}	4.10 ± 0.16 ^{d}	2.64 ± 0.20 ^{d}
JEF 10	10.89 ± 0.45 ^{b}	94.32 ± 1.03 ^{ab}	-0.73 ± 0.07 ^{b}	8.06 ± 0.45 ^{c}	6.89 ± 0.50 ^{c}
JEF 20	11.26 ± 0.44 ^{b}	93.36 ± 0.61 ^{b}	-1.12 ± 0.13 ^{a}	12.09 ± 0.52 ^{b}	11.15 ± 0.57 ^{b}
JEF 30	12.47 ± 0.56 ^{a}	93.44 ± 0.34 ^{b}	-1.22 ± 0.24 ^{a}	14.81 ± 0.38 ^{a}	13.60 ± 0.46 ^{a}

Note: Control film (CF) and films incorporated with 10, 20 and 30% (v/v) of jatobá pulp, named JEF10, JEF20, and PJEF30, respectively. Mean ± standard deviation. Different letters in the same column indicate significant differences ($p < 0.05$) by Tukey's test.

The incorporation of JPE into the biopolymer matrix resulted in concentration-dependent changes in the optical properties of the films. The opacity and ΔE increased with increasing extract concentration, whereas luminosity decreased numerically (Table 1). However, the Tukey test indicated that the luminosity of JEF10 did not differ significantly from that of the control film (CF), whereas JEF20 and JEF30 showed significantly lower L values than the control. Opacity increased from 9.86% in CF to 12.47% in JEF30, indicating a greater reduction in light transmission through the films as the extract concentration increased.

The increased opacity and ΔE can potentially provide greater protection against photodegradation of packaged foods [45]. Nevertheless, opacity measurements alone do not establish the extent of protection against photodegradation, which also depends on the wavelength-dependent light transmission and the susceptibility of the packaged food. Therefore, the potential protective effect of the films should be interpreted as a possible application rather than as a demonstrated preservation effect.

Regarding the chromatic coordinates, a* values became progressively more negative as the extract concentration increased, changing from − 0.24 in CF to − 1.22 in JEF30. According to the Tukey test, JEF20 and JEF30 did not differ significantly from each other for this parameter, although both differed from JEF10 and CF. In contrast, b* increased from 4.10 in CF to 14.81 in JEF30, with significant differences among all four formulations. These changes indicate a shift toward greener tones along the a* axis and an increasingly pronounced yellow component along the b* axis. According to Alves-Silva, Romani, and Martins [3], jatobá pulp can exhibit coloration ranging from yellow to light green. Thus, the changes in a* and b* are consistent with the incorporation of the naturally colored extract.

The ΔE values increased from 2.64 in CF to 13.60 in JEF30, with significant differences among all formulations. This progressive increase indicates a greater color difference relative to the reference used in the color-difference calculation as the extract concentration increased. The observed changes in opacity, luminosity, and chromatic coordinates therefore demonstrate that JPE incorporation modified the visual characteristics of the films, with more pronounced changes at higher extract concentrations.

In general, the films exhibited low opacity and high luminosity. These optical characteristics may be advantageous when a light-colored appearance is desired; however, the suitability of a particular color and opacity depends on the intended product and packaging function. Although increased opacity may reduce light exposure, the color of a packaging material can also influence the visual perception of the packaged food [15]. According to Nogueira and Martins [30], the film color should be compatible with the packaged product to maintain its image and perceived quality, which may be facilitated by greater packaging transparency. For soluble sachets intended for powdered foods, the balance between visual appearance and potential light protection should therefore be considered alongside the mechanical, solubility, and water vapor barrier properties discussed previously.

3.2 Effects of active alginate sachets on milk powder quality

The quality of instant whole milk powder (WMP) is assessed using physicochemical and reconstitution parameters established by Brazilian regulations. According to RDC No. 53/2018 [9], relevant requirements include a minimum solubility of 85%, a minimum fat content of 26%, and a maximum moisture content of 5%. The regulation also establishes a limit for titratable acidity, expressed as 1.8 mL of 0.1 N NaOH per 100 g of solids. These parameters provide a basis for evaluating the quality of the powder during storage. However, the results of each analysis must be interpreted according to its specific method and unit of expression.

The physicochemical properties of milk powder influence its behavior during storage, handling, and reconstitution [17]. Its stability is affected by several factors, including the quality of the raw milk, processing conditions, exposure to oxygen and light, moisture content, and storage temperature. Packaging therefore plays an important role in limiting environmental exposure and preserving product quality [26, 37]. The effectiveness of a packaging system, however, depends on the combined performance of the material, the package structure, and its closure under the conditions in which the product is stored.

Water-soluble sachets are a potential alternative for applications in which the packaging can be introduced directly into the reconstitution water, reducing the need to remove and discard the package before use. In addition, active packaging incorporating plant-derived compounds has been investigated as a strategy to delay food oxidation [5]. In this study, sodium alginate sachets without extract and sachets incorporating jatobá pulp extract were evaluated for their effects on WMP quality during 50 days of storage at 30°C. Milk powder stored in synthetic polypropylene sachets was included for comparison. The parameters evaluated included thiobarbituric acid-reactive substances (TBARS), titratable acidity, pH, moisture content, color, and reconstitution characteristics.

3.2.1 *TBARS, titratable acidity, pH, moisture and color of milk powder stored in sachets*

Whole milk powder contains a substantial lipid fraction and is susceptible to lipid oxidation during storage [27]. The TBARS assay is commonly used to assess the accumulation of thiobarbituric acid-reactive substances associated with oxidative deterioration. However, the assay is not specific to a single oxidation product; its results should therefore be interpreted as an indicator of TBARS rather than as a direct measurement of one particular compound [26]. Figure 4 presents the TBARS values (A), Acidity titratable (B), pH (C), moisture (D) and *Color* (E) of WMP stored in synthetic sachets (SS), control soluble alginate sachets (CS), and soluble alginate sachets containing jatobá extract (EJS) during 50 days of storage.

Mean ± standard deviation. Different letters in the same image indicate significant differences ($p < 0.05$) by Tukey's test

The highest TBARS values were observed in WMP stored in synthetic sachets, followed by the control soluble sachets, whereas the jatobá-extract sachets showed lower values. This pattern suggests that the incorporation of jatobá extract into the alginate sachets may have contributed to limiting the accumulation of TBARS during storage. This effect may be related to the antioxidant activity previously observed in the jatobá-containing films, particularly the presence of phenolic compounds that can participate in reactions involved in the inhibition of lipid oxidation. Similar effects have been reported in previous studies investigating antioxidant strategies for milk powder preservation. Granda-Restrepo et al. [21] investigated multilayer active packaging incorporating α-tocopherol and reported its effectiveness in delaying lipid oxidation in whole milk powder. Likewise, Tzima et al. [48], investigated the antioxidant effects of rosemary-derived compounds in full-fat milk powder stored under accelerated conditions (40°C and 23% relative humidity) for 90 days, reporting a reduction in TBARS values in the samples treated with rosemary antioxidants. Although these studies used different antioxidant compounds and preservation strategies, their findings support the potential of incorporating antioxidant agents into packaging systems or milk powder formulations to reduce lipid oxidation. In this context, the lower TBARS values observed in WMP stored in the jatobá-extract sachets suggest that this packaging system may offer an alternative approach to improving oxidative stability during storage.

The titratable acidity and pH results are presented in Fig. 4B and 4C, respectively. The initial acidity was reported as 1.22%, expressed as lactic acid. During storage, WMP packaged in sachets containing jatobá extract exhibited lower acidity, whereas the synthetic sachets showed higher acidity at the end of the 50-day period. This pattern indicates that the packaging treatments differed in their effect on the acidity measured in the samples. The pH analysis can also be an indicator of quality and milks thermal stability. The formation of organic acids resulting from oxidation can lower the milk's pH because organic acids release hydrogen ions into the solution, increasing the concentration of these ions and consequently reducing pH [19, 20].

The results observed are in line with previous research on active packaging systems containing antioxidant compounds. Alves-Silva et al.[5] reported delayed lipid oxidation in olive oil stored in alginate

sachets containing macaúba-derived compounds. Granda-Restrepo et al. [21], investigated multilayer packaging containing α-tocopherol for milk powder, while De Abreu et al. [1], evaluated antioxidant-containing low-density polyethylene films for frozen Atlantic salmon, and although these studies involve different foods, packaging materials, and active compounds, they provide context for the potential of antioxidant packaging to delay oxidative changes. In the present study, the lower TBARS values observed with the jatobá-extract sachets, together with the acidity and pH results, support further investigation of this formulation as an active packaging system for WMP.

Moisture content is an important quality parameter in WMP because water uptake can alter powder flowability and physical stability and may affect reactions occurring during storage [19, 26, 37]. As shown in Fig. 4D, the powder stored in synthetic sachets had lower moisture content than the powder stored in the control and jatobá-extract soluble sachets. Nevertheless, all treatments remained within the maximum moisture content of 5% specified by RDC N°. 53/2018[9] during the 50-day storage period.

The moisture results are particularly relevant when considered alongside the TBARS and acidity findings. Although the synthetic sachets were associated with lower moisture content, they showed higher TBARS values and higher acidity at the end of storage than the jatobá-extract sachets. Thus, moisture content alone does not explain the differences observed in the oxidative and acidity indicators. The results suggest that other factors, including the presence or absence of antioxidant compounds and differences in the packaging systems, may have contributed to the observed pattern.

Color is another parameter relevant to the quality of WMP during storage. Changes in color may be associated with several processes, including oxidative reactions and nonenzymatic browning, although color measurements alone cannot identify the mechanism responsible for a change [26, 52]. The chromatic coordinate b* represents the yellow–blue axis, with positive values indicating a shift toward yellow. It is therefore useful for describing changes in the characteristic yellowish appearance of milk powder [32]. The b* values obtained during storage are shown in Fig. 4E. All treatments retained the yellowish appearance characteristic of bovine milk powder, which is partly associated with fat-soluble pigments such as carotenoids [43]. The b values decreased during the first 28 days of storage, increased at day 35, and subsequently decreased toward values closer to those measured at the beginning of the experiment. This non-linear pattern indicates that color did not change progressively in a single direction throughout storage. Similar temporal variation has been reported in research on milk powder storage[26], although the specific causes of the changes should not be assumed to be identical across studies.

Upon reaching 50 days of storage, a decrease in the b* value was observed in the sachet incorporated with jatobá extract. It is important to note that packaging materials can chemically interact with the components of milk powder, which can affect the pigments responsible for the yellow color, resulting in a decrease in b* values. The characteristic color of jatobá pulp can vary from yellow to light green [3]. Therefore, this reduction may be related to the migration of color from the packaging to the milk powder.

3.2.2 Reconstitution characteristics of milk powder

Dispersibility and the insolubility index (ISi) are important parameters for evaluating the reconstitution characteristics of WMP. Dispersibility describes the ability of powder particles to separate and become distributed in water, whereas the insolubility index reflects the insoluble residue remaining after reconstitution under specified test conditions [46]. Dispersibility ranged from 92.01% to 92.40%, and the values were above the minimum of 85% specified for instant milk powder under RDC No. 53/2018 [10]. The results are presented in Table 2, the values reported indicating no statistically significant differences among the listed dispersibility measurements according to the indicated Tukey test. Within the conditions evaluated, storage in the different sachets did not produce a detectable change in dispersibility.

Table 2
Reconstitution characteristics of whole milk powder storage in sachet for 50 days

Days	Amostra	dispersibilidade	Insolubility index (g)
0	CM	92.02 ± 0.01^{a}	0,21 ± 0.01 ^{a}
	SS	92.29 ± 0.23 ^{a}	0,17 ± 0.01 ^{b}
7	CS	92.40 ± 0.18 ^{a}	0,17 ± 0.01 ^{b}
	EJS	92.17 ± 0.12 ^{a}	0,15 ± 0.01 ^{b}
	SS	92.05 ± 0.01 ^{a}	0,19 ± 0.01 ^{a}
50	CS	92.01 ± 0.01 ^{a}	0,14 ± 0.01 ^{b}
	EJS	92.04 ± 0.03 ^{a}	0,15 ± 0.01 ^{b}

CM: control milk powder; SS: synthetic sachets; CS: control soluble sachets; EJS: jatobá-extract soluble sachets. Values are expressed as mean ± standard deviation. Different letters within the same comparison indicate significant differences ($p < 0.05$) according to Tukey's test.

In the present study, the dry residues in powder (ISi) ranged from 0.21 to 0.14 mg. Based on the results obtained, it was observed that none of the packaging affected the reconstitution characteristics of whole milk powder. It is important to highlight that an instant product should have the ability to mix easily and dissolve rapidly in cold or hot water, forming a homogeneous solution free from lumps. Therefore, solubility tests of the sodium alginate sachets, with and without the addition of extract, were also performed (Fig. 5). As shown in Fig. 5A and Fig. 5B, the sodium alginate sachets (CS and JES) containing milk powder were immersed in water at the temperatures of 37 and 50°C to demonstrate their solubility. At both temperatures, complete dissolution of the sachets was apparently observed. However, at the temperature of 50°C less alginate residue resulted after filtering the samples (Fig. 5C). Furthermore, storage time did not influence the solubility of the sachets. Such results suggest that a

higher temperature may be more suitable for using these sachets in terms of efficiency and the quality of the final product, i.e., the reconstituted milk.

4 Conclusion

The present study has revealed promising results regarding the incorporation of jatobá pulp extract into flexible alginate films. This addition led to an increase in tensile strength, elongation at break, and opacity of the films, as well as a reduction in water solubility, demonstrating its potential for the production of stronger and more effective packaging. It was also observed that an increase in the extract concentration did not significantly enhance the antioxidant activities of the films. The soluble sachets, especially those containing active jatobá extract, showed the ability to minimize the effect of lipid oxidation, reduce pH variations, and control the acidity index of milk powder. These results clearly indicate the feasibility of using soluble sachets as a promising solution for storing sensitive products like whole milk powder.

The results of this study have provided valuable insights into the feasibility and benefits of using a soluble film as a sachet for milk powder. This approach can offer advantages in terms of convenience for consumers while maintaining product quality and durability. Additionally, the consideration of environmental aspects is crucial as it contributes to sustainability and environmental responsibility in the food packaging industry.

Declarations of 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.

Declarations

Author Contribution

Gisele Fernanda Alves-Silva: Conceptualization, Methodology and Writing Original draft preparation. Viviane Patrícia Romani: Supervision, Writing, Reviewing and Editing. Vilásia Guimarães Martins: Conceptualization, Supervision, Writing and Reviewing.

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Figures

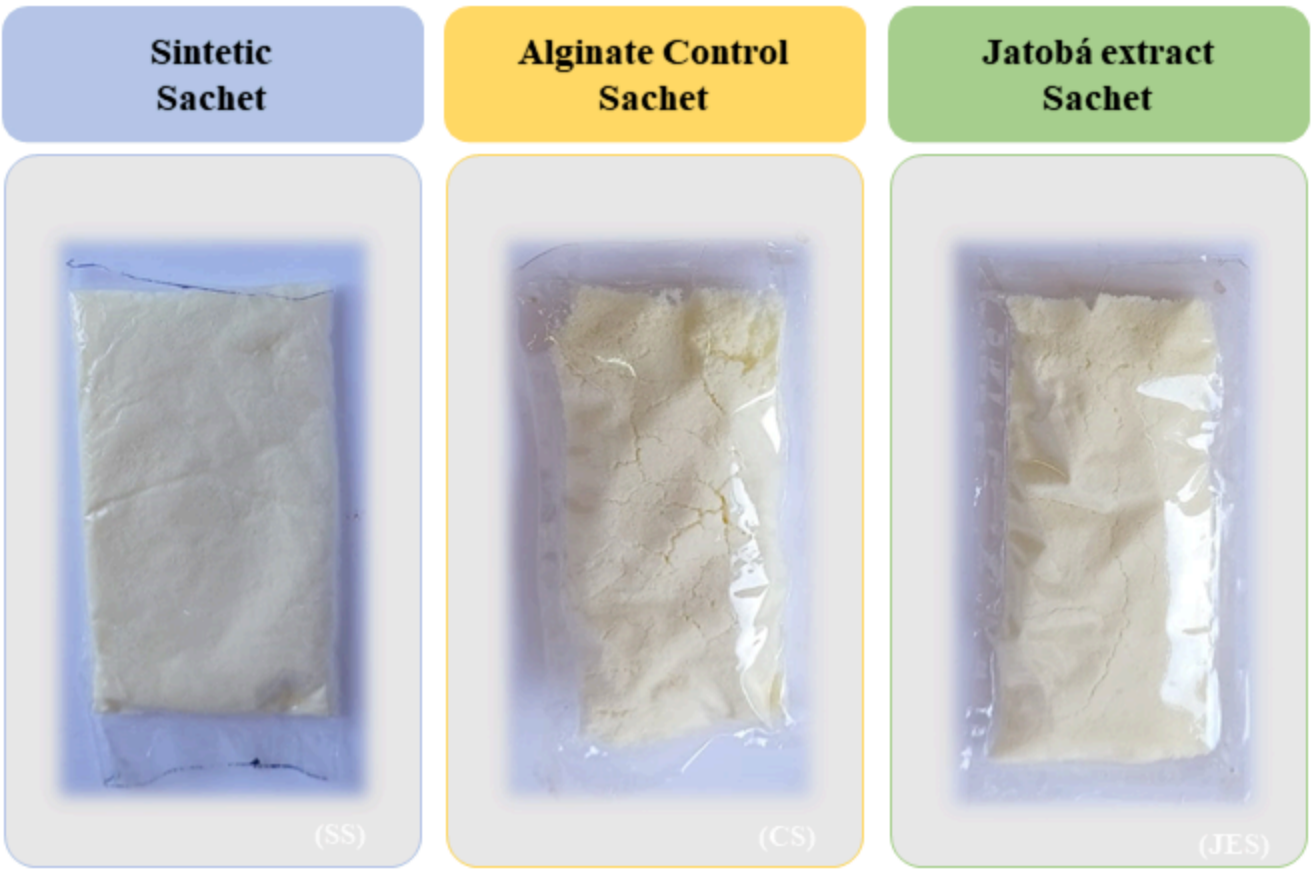


Figure 1

Synthetic (SS) and soluble (CS and JES) sachets containing whole milk powder

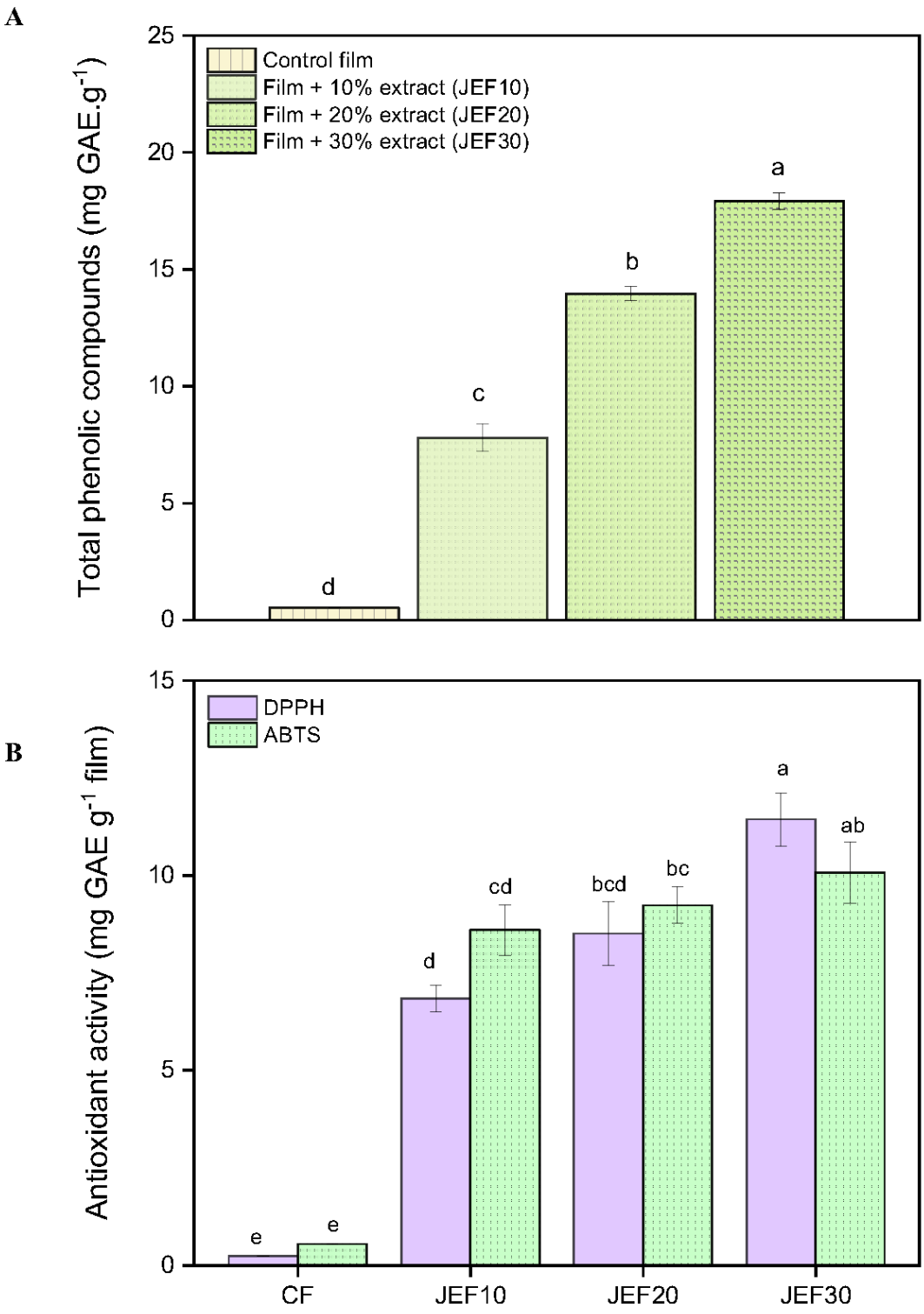


Figure 2

Total phenolic content (TPC), (A) and antioxidant activity determined by the ABTS•+ and DPPH assays (B) of sodium alginate films containing different concentrations of jatobá pulp extract.

Mean ± standard deviation. Different letters in the same image indicate significant differences ($p < 0.05$) by Tukey's test

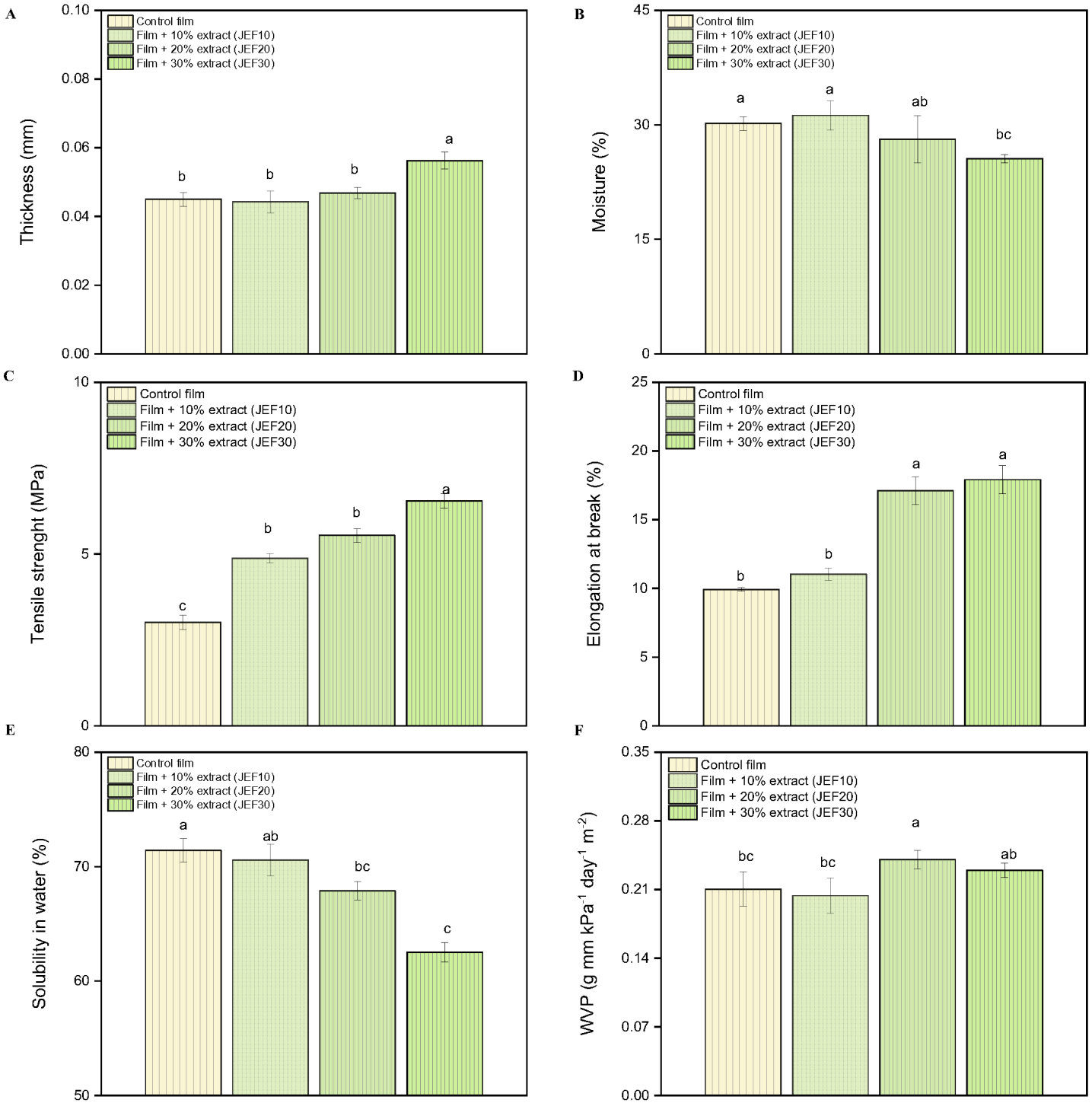


Figure 3

Physical, Mechanical and barrier properties of sodium alginate films with and without the incorporation of jatobá pulp extract

Mean ± standard deviation. Different letters in the same image indicate significant differences ($p < 0.05$) by Tukey's test

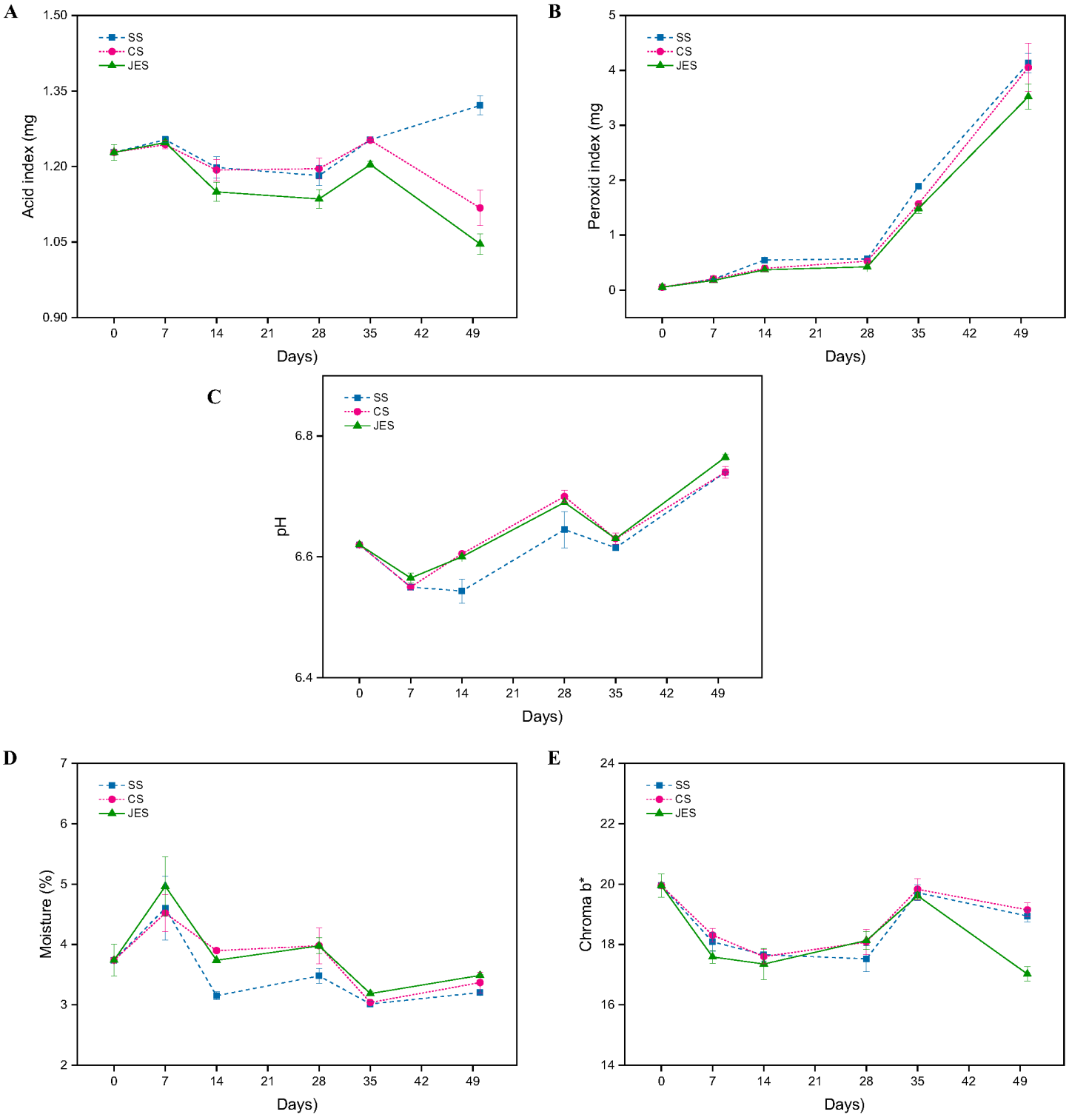


Figure 4

TBAs (A), Acidity titratable (B), pH (C)and moisture (D) of the milk powder storage in sachets for 50 days. SS: synthetic sachets, CF: Control sachet soluble and EJS: jatobá extract sachet soluble.

Mean ± standard deviation. Different letters in the same image indicate significant differences ($p < 0.05$) by Tukey's test

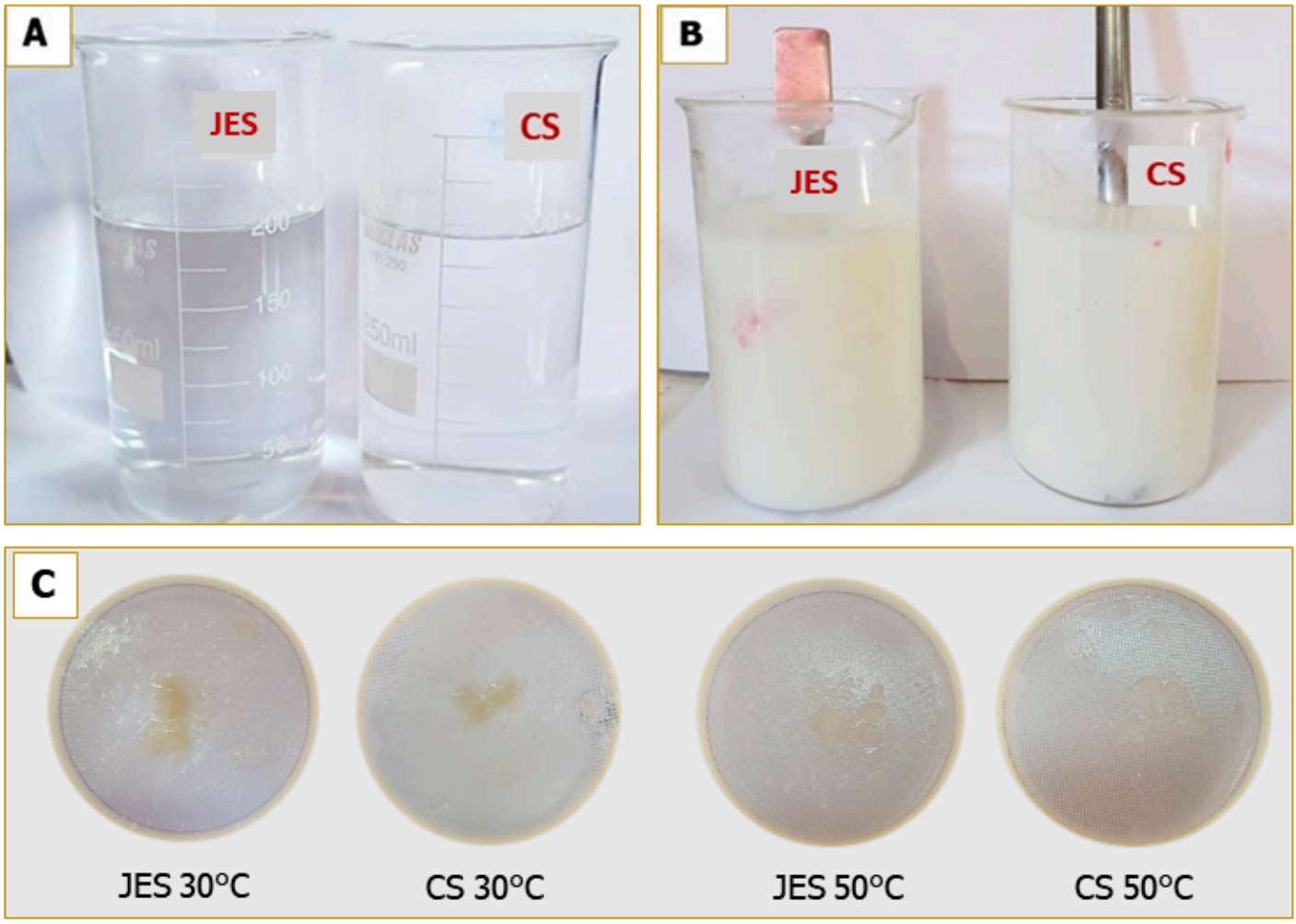


Figure 5

Dissolution of sodium alginate sachets containing whole milk powder at 37 and 50 °C.

Supplementary Files

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