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Development and characterization of active edible *Opuntia* mucilage/locust bean gum packaging incorporated with *Opuntia* cladodes polyphenols

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ABSTRACT

The food packaging has witnessed advancements recently as a response to necessity to minimize harmful plastic food waste. An active edible packaging was designed by blending 2 % of *Opuntia* cladode polyphenols with a hydrocolloid matrix made of their mucilage and Locust bean gum "LBG". The yield of mucilage and gum extraction was 1.35 % and 58.99 %. The active film CL-LBG+ was sensorially preferred for its surface softness and caramel flavor. It was thicker, moisturous and transparent, along with a lower hydrosolubility and water vapor permeability (71.21 % and 2.97 × 10^{-6} $g.m^{-1}.s^{-1}.Pa^{-1}$). The active film was five times less dense and twofold hydrophobic than the control film CL-LBG. The functional groups of carbohydrate moieties constitutive of LBG and mucilage, on top of those of glycerol and polyphenols, were identified by ATR-FTIR and NMR. The biological potency of films, the antioxidant activity was up to 49.79 % after incorporation of polyphenols. A positive boosting was noted for the antiradical efficacy in fat food simulant over aqueous simulant. The freshness of active-coated apple slices refrigerated for 9 days was preserved at the marketing threshold, consequently of slowed ripening mechanism and metabolic intensity amortizing their weight loss and pH regression, at a steadier total soluble solids level.

1. Introduction

The plant realm constitutes an unparalleled reservoir of secondary metabolites evolved through millennia of life adaptation. This remarkable complex array of bioactive compounds has been used by humans since antiquity in therapeutic, technological, and pharmaceutical applications. The food sector exemplifies this versatile exploitation of plant capital, where growing global demand imposes not only increased production but also advanced preservation technologies to maintain year-round availability while preserving nutritional, hygienic, and sensory qualities (Thiviya et al., 2023; Gamage et al., 2024). These challenges have driven the development of sustainable bio-packaging solutions that simultaneously address food safety, environmental concerns, and value-addition through the functional enrichment of food products (Gheribi et al., 2019; Rodríguez et al., 2020; Gamage et al., 2022; Liyanapathiranage et al., 2023). Whether used as edible films or coatings, natural polymers have been widely investigated for this purpose. Since they are included in this molecular category, the highly branched hetero-polysaccharide hydrocolloids like *Opuntia* cactus mucilage and *Ceratonia siliqua* L. locust bean gum "LBG" are attracting increasing interest for their chemical composition, structural features, and biotechnological applications, which open new opportunities in behalf of the food packaging industry (González Sandoval et al., 2019; Moussaoui et al., 2022, Yun et al., 2022). Despite the promising

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properties of *Opuntia* mucilage or LBG as economically profitable material due to their availability, low cost and efficiency when used as primary packaging for food products, other enhancements are required to reinforce their poor barrier and mechanical properties and to grant better biological potency (Gheribi et al., 2019; González Sandoval et al., 2019). Likely, the unification of biopolymers with different structures alleviates the inherent disadvantages following the use of individual polymers and can ameliorate packaging film properties owing to the presence of strong intermolecular interactions (Kanatt, 2020; Moussaoui et al., 2022). Additionally, the incorporation of bioactive compounds such as polyphenols in biobased films may improve their bioactive characteristics, especially their antimicrobial and antioxidant activities when compared to standalone films, which avoid color and texture changes or the creation of off-odors and off-flavors, prompting ultimately consumer choice and acceptance (Aragón-Gutiérrez et al., 2020; Rodrigues et al., 2023). The ultimate aim of this study was to enhance the shelf life of food products while concurrently minimizing environmental impact, aligning with the current context of reducing food waste and sustainability. This work focuses on developing, characterizing and applying an original edible active packaging film derived from the crosslinking of *Opuntia* cladodes mucilage and Locust bean gum hydrocolloids sourced from the region of Tissemsilt-Algeria (south west of Algiers). The active component of the film will be represented by the polyphenols extracted from the same cladodes. The resulting active film, plasticized with glycerol, will be applied as a coating on fresh apple slices and assessed for its efficacy in extending their shelf life at a refrigerated temperature.

2. Materials and methods

2.1. Materials

A random collection of mature cladodes of 30 to 50 cm in length and 15 to 30 cm in width was carried out in February 2022 from many living inermis Opuntia ficus indica plants at Laayoune location (41″ North, 1° 59′ 53″ East), 200Km in South West of Algiers -Algeria. The samples were cleaned of thorns, disinfected with 10 % sodium hypochlorite, and rinsed with distilled water for mucilage extration, while a fraction was dried totally at 40 °C before being ground into a fine powder to be used later for polyphenol extraction. Mature carob pods were harvested randomly from trees in the area of Lardjem at Tissemsilt-Algeria (35° 44′ 58″ north, 1° 32′ 54″ east) in July 2022. After grinding the pods, recovered seeds were stored dry in polyethylene bags at room temperature until use. Fresh, intact apples without deterioration or injury were brought from the local market. Ascorbic acid, Calcium chloride "$CaCl_2$" (≥97 %), 2,2-Diphenyl-1-picrylhydrazyl "DPPH", ethanol, Methanol, gallic acid, sodium carbonate "Na_2CO_3" (≥99.0 %), glycerol (92.09 g/mol) were purchased from Aldrich (Sigma–Aldrich, St. Louis, MO, USA).

2.1.1. Extraction of hydrocolloids

Intact peeled cladodes cut into cubes were cooked for 3 to 5 min in a microwave oven (Samsung, ME733K, South Korea) (900 W) until tenderness. The mucilage was extracted by blending the cubes and centrifuging the mixture (8000 rpm/15 min) at 4 °C followed by a drying step at 60 °C for 72 h (Du Toit & De Wit, 2011). 100 g of locust bean seeds were immersed in 800 ml of distilled water at 100 °C for 1 h. The germ-free endosperm obtained after discarding the swollen teguments was recovered and oven-dried (100 °C/1 to 2 h) to constant weight. After that, the dry matter crushed and sieved, representing the crude carob gum was stored in airtight boxes at room temperature until utilization (Farahnaky et al., 2014).

2.1.2. Extraction and quantification of Opuntia cladode polyphenols

10 g of cladodes powder was macerated for 24 h in a methanol-water mixture (1.3/8.7: V/V) at a ratio of 1/10 (W/V), with occasional agitation. The macerate was filtered then stripped of solvent by rotavap evaporation (Heidolph, Laborota 4000, Germany) at 40 °C under reduced pressure. The final dry extract was weighed, reconstituted in 5 mL of distilled water and stored in darkness and airtight conditions (Merghem et al., 1995). Total polyphenols were assessed by adding 1 mL of polyphenol extract to 1 mL of 10-fold diluted Folin-Ciocalteu reagent. After 4 min, 8 mL of 7.5 % (W/V) sodium carbonate Na_2CO_3 was poured with a concomitant gentle stirring. Subsequently, the mixture was incubated for 2 h in the dark at room temperature and was subject to an immediate measurement of absorbance at 765 nm using a UV–VIS spectrophotometer (Jenway™ 7315, Stone-Staffordshire, United Kingdom). The equation of the calibration curve established with gallic acid and expressed in micrograms of gallic acid equivalents per gram of cladode dry matter (mg GAE/g DM) was used to calculate the concentration of total polyphenols (Singleton et al., 1999).

2.2. Film processing

2.2.1. Film-forming dispersions

Film-forming solutions were prepared by gradually dissolving 2 g of mucilage in 200 ml of distilled water under constant stirring for 3 h at room temperature. Right after, 2 g of LBG was added progressively to the mucilage solution and stirred in parallel for 1 h at 25 °C. The plasticizer glycerol was then added at a rate of 40 % (w/w) based on the total weight of the hydrocolloids. Cladode polyphenols were added at 2 % (w/v) of the film-forming solution. The resulting viscous solution (CL-LBG+) underwent a final homogenizing agitation at 70 °C/30 min. The solution without polyphenols represented the control of the study (CL-LBG).

2.2.2. Film preparation

According to practical optimization, film-forming solutions were poured into silicone molds in a proportion of 1.02 mL/cm2 and dried in an oven at 40 °C for 48 h. Films recovered and cooled in a desiccator (25 °C, 53 % Relative Humidity RH) for 48 h were stored in airtight bags until characterization as reported by Gheribi et al. (2019).

2.2.3. Characterization of edible biofilms

2.2.3.1. Sensory evaluation of films. The physical appearance, color, smell, taste and overall acceptability of the films were evaluated.

2.2.3.2. Film thickness and density. The film's thickness represented the average value of ten measurements at different points using a micrometer (Fowler, Newton, Massachusetts, USA) (Gheribi et al., 2019). Consequently, the density of films was measured using the technique of Ramos et al. (2012). A 2 cm^2 piece of film (s), whose thickness (e) was determined beforehand, was weighed (m), and the density was calculated as follows (Li et al., 2020):

$$d = \frac{m}{s.e}$$

2.2.3.3. Moisture content and water solubility. The relative humidity (RH) content of films was determined according to the protocol of Jouki et al. (2013). Samples of 2 cm^2 were weighed (m_i), oven-dried at 90 °C for 24 h and then reweighed (m_f) after cooling in a desiccator to room temperature. Moisture content was determined according to the equation of Gheribi et al. (2019):

$$HR = \frac{mi - mf}{mi} \times 100$$

Films water solubility (W_s) was tested by immersing the previous dried samples in 50 ml distilled water at 25 °C for 30 min. The undissolved fragments were dried (90 °C/24 h) and reweighed (m_F) again after cooling to room temperature (Jouki et al., 2013). The water solubility was calculated according to the following formula:

$$Ws = \frac{mi - mf}{mi} \times 100$$

2.2.3.4. Water contact angle. This test reflects the hydrophobicity of the film surface. The water contact angle (°) or the angle between the baseline of a 4 µL distilled water droplet deposited upon the film surface and the tangent line at the point of contact was provided by the goniometer (Pocket goniometer PGX, Sweden). The mean value of five reads at different positions on the film surface was calculated for each film type (Gheribi et al., 2019).

2.2.3.5. Water vapor permeability (WVP). This attribute was determined by sealing an acrylic cup containing the hygroscopic $CaCl_2$ with the films. With its weight already noted, the assembly was placed in a chamber containing 500 ml of distilled water (20 °C) to maintain the relative humidity inside at around 99 % during the experiment. The weight gain of the cups was measured for 6 h at an interval of 1 h to obtain the water vapor transmission rate (WVTR) of the films (González Sandoval et al., 2019). The WVP was calculated as follows:

$$WVP(\text{g.m} - 1.\text{s} - 1.\text{Pa} - 1) = \frac{WVTR}{A}\frac{e}{\Delta Pv}$$

Where:

WVTR: the water vapor transmission rate ($g.s^{-1}$) or the slope of the linear regression [weight gain = f (time)], calculated by plotting the final weight minus the initial sample weight (w_f - w_0) as a function of time (t); e: average film thickness (m); A: transfer area (m^2); ΔPυ: equals to 2337 Pa, is the water vapor pressure difference between the $CaCl_2$ atmosphere and the chamber atmosphere.

2.2.3.6. Film transparency. According to the method of González Sandoval et al. (2019), the absorbance and transmittance of (0.5 cm x 4.0 cm) edible film strips placed in a quartz cell were measured at 600 nm using a UV–visible spectrophotometer (Jenway™ 7315, Stone-Staffordshire, United Kingdom). Transparency is calculated using the ratio:

$$Transparency = \frac{A600}{S} = \frac{-log\ T600}{S}$$

Where:

A_{600} and T_{600}: absorbance and transmittance at 600 nm, respectively; s: film thickness.

2.2.3.7. ATR-FTIR spectroscopy. The ATR-FTIR spectra of CL-LBG and CL-LBG+ films were registered with Agilent Cary 630 FTIR spectrometer (CA, USA) at room temperature between 4000 and 600 cm^{-1} using 8 scans at a resolution of 2 cm^{-1}.

2.2.3.8. NMR analysis of films. ^{13}C and ^{1}H NMR analysis was carried out using Bruker Avance 500 MHz spectrometer (Bruker Avance, Rheinstetten, Germany) at 125 and 500 MHz frequencies. Analyses were recorded at room temperature in D_2O (^{1}H spectrum: 64 scans, 3.1 s acquisition time; ^{13}C spectrum: 100,000 scans, 1.1 s acquisition time).

2.2.4. Film activities

Film sections of 2 cm^2 were wholly dissolved in 10 mL of distilled water in darkness by stirring at 100 rpm/25 °C (Kanatt, 2020; Valdés et al., 2021).

2.2.4.1. Total phenolic content (TPC) and antioxidant activity (DPPH inhibition). Determining total phenolic content (TPC) in active films followed identical protocol detailed previously (Singleton et al., 1999). Meanwhile, the antioxidant capacity of films with or without polyphenols was assessed by measuring DPPH radical scavenging activity according to the method of Kanatt (2020). Briefly, 100 µL of each solution was mixed with 3.9 ml of a 0.06 mM methanolic solution of DPPH in a closed cuvette. After vigorous shaking, the mixture was left in the dark for 30 min at room temperature. Absorbance was measured at 517 nm against a negative control, substituting the different samples with distilled water. The anti-free radical activity is the decrease in DPPH absorbance, and the percentage inhibition was calculated according to the following equation (Aragón-Gutiérrez et al., 2020):

$$I(\%) = \left(\frac{Ac - As}{Ac}\right).100$$

Where:

I: DPPH inhibition rate (%), Ac: absorbance of control, As: absorbance of samples.

2.2.4.2. Antioxidant activity in food simulants. The antioxidant activity of CL-LBG+ was evaluated using the DPPH method in 10 % and 95 % ethanol (v/v) as an aqueous and fatty food simulants (Aragón-Gutiérrez et al., 2020). These two simulants mimic the behavior of real aqueous and fatty foods. 10 % ethanol is assigned for foods that have a hydrophilic character and are able to extract hydrophilic substance, while 95 % ethanol is assigned for foods that have a lipophilic character and are able to extract lipophilic substances (European Commission, 2011). Briefly, 3 cm^2 of each film was dipped in 5 mL of the simulant (surface/volume ratio ~ 6 dm^2/l). After 1, 6, 18 and 24 h, aliquots of 100 µL of each mixture film/simulant were mixed with 3.9 mL of 0.06 mM of DPPH methanolic solution. The mixture was shaken vigorously and kept in the dark at room temperature for 30 min. The absorbance was measured (517 nm) against a negative control containing pure ethanol at the place of dissolved films, while ascorbic acid was the positive control in the same concentration range as films (Aragón-Gutiérrez et al., 2020; De Souza et al., 2020). The DPPH inhibition efficacy is expressed as follows:

$$IE\% = 100\ .\ \frac{A(\text{Control}) - A(\text{sample})}{A(\text{Control})}$$

Where:

IE %: percentage of DPPH inhibition, $A_{(control)}$: absorbance of the control, $A_{(sample)}$: absorbance of the film extract.

2.2.5. Application of active films CL-LBG+ as packaging coatings

The exact film-forming solutions prepared previously, but exempt from plasticizer, were used as coating solutions. Fresh, intact apples were cut into identical pieces of approximately equal weight. Three groups of 6 slices were soaked for 30 s in CL-LBG, CL-LBG+ and water (control). Subsequently, the superfluous coating solution was drained and the coated apple slices were dried at room temperature for 30 min. The fruits were stored separately in a 4 °C refrigerator for 9 days.

2.2.5.1. Effect of coating on weight and pH of apples. Fresh and post-storage weights of apple pieces for each group were taken by an analytical balance. The pH values of all pieces were measured as well by dipping the pH meter electrode into a 10 % (w/v) solution obtained by soaking apple pieces in distilled water for 5 min (Cunniff, 2000).

2.2.5.2. Total soluble solids (°Brix). The content of soluble solids was assessed using a refractometer (Optika, Ponteranica, Italy) (°Brix), depositing a drop of juice obtained after grinding the coated or the uncoated slices on its prism (Cunniff, 2000).

2.2.5.3. Sensory assessment. Sensory attributes, including taste, odor, color, texture and overall acceptability of coated and uncoated apples, presented monadically to consent untrained panelists, were determined according to the 9-point hedonic scale: (9) I like Extremely, (8) I like very much, (7) I like moderately, (6) I like slightly, (5) Neither liked nor

hated, (4) I hate slightly, (3) I hate moderately, (2) I hate very much, (1) I do not like it at all (Rodríguez et al., 2020).

2.3. Statistical analysis

Data from triplicate measurements were expressed as means ± standard deviation (SD). Statistical analyses were conducted using Student's *t*-test and analysis of variance (ANOVA) in IBM SPSS Statistics (version 26). Differences were considered statistically significant at $p < 0.05$.

3. Results and discussion

3.1. Extraction yields

Table 1 brings together the extraction yields (on a dry matter basis) of the two colloids mucilage and LBG, in addition to cladodes polyphenols:

3.1.1. Hydrocolloids yields

The yield of mucilage in the cladodes of Tissemsilt's Opuntia ficus indica was equal to 1.36±0.04 % (Table 1), which represents a lesser percentage than that of 2.18±0.44 % reported in our previous work; Moussaoui et al. (2022), for cladodes of the same district but located in a higher mountainous location endowed with different climate. The mucilage content depends extremely on cactus species, organ, age or maturity, climatic conditions and harvest period (Gheribi et al., 2019). Sáenz et al. (2004) considered the intensification of mucilage production as a protective reaction to the plant against abiotic hostilities such as drought. On the other hand, some extraction's influencing factors, such as powder particle size, solvent nature and temperature, had a noticeable impact on extraction performance and, thereby, on mucilage levels (Golstein & Nobel, 1991; Gheribi et al., 2019). The extraction yield of crude carob gum of Laayoune region trees reached 59.81±1.73 % (Table 1), a higher level than 53.88±1.84 % noted by Moussaoui et al. (2022) for another Algerian carob, and a figure included in the range 51 to 61 % reported by Dakia et al. (2008). In fact, the recovery of carob gum from the seeds is related to the extraction method, cultivars, origin and the growing conditions of the carob (El Batal et al., 2013).

3.1.2. Polyphenol extraction yield

As shown in Table 1, the extraction yield of polyphenols from Opuntia cladodes touched the bar of 5.42±0.30 %. When compared to Mexican nopals, the cladodes of Tissemsilt had a less polyphenols synthesis capability (180 vs 63.51±2.06 mg GAE/g DM) (Gallegos-Infante et al., 2009). Nopals analyzed by Medina-Torres et al. (2011) didn't exceed 60 g/kg. According to Méndez et al. (2015) and Rocchetti et al. (2018), the noted values were 128.8 ± 29.4 (mg/100 g) for Spanish cladodes and 2633.10 ± 214.78 (mg GAE/kg FW) for Italian cladodes. However, the noted value is greater than the concentration of 39.26 mg GAE/g DM given by De Santiago et al. (2021). Generally, the fineness of cladodes powder and its drying temperature not exceeding the threshold of 40 °C prevent the degradation of polyphenols and offer a greater contact surface, facilitating the penetration of the solvent into the plant matrix and the dissolution of the target molecules. The significant variation reported in the results of different research is partly due to the variation in the methodology applied (De Santiago et al., 2021). In addition, several studies have shown that maceration is the best way to extract polyphenols, where the sample/solvent ratio, extraction time and temperature directly affect the extraction yield (Chaalal et al., 2012; Kechebar et al., 2017; Koné et al., 2017). The multitude of results expressions (fresh weight or dry matter, millilitre of extract) is another source of ambiguity making their comparison a more or less difficult task (De Santiago et al., 2021). Last but not least, the noticeable differences of total phenolic amount in Opuntia cladodes reported in literature was highly correlated to the variety and species, the climatic conditions, and the maturity stage (Guevara-Figueroa et al., 2010; Blando et al., 2019)

Table 1
Yields of Opuntia mucilage, LBG and cladodes polyphenols.

Hydrocolloids		
	Yield (%)	
Opuntia mucilage	1.36±0.04	
Carob gum	59.81±1.73	
Polyphenols		
	Content (mg GAE/g DM)	Yield (%)
Opuntia cladode polyphenols	63.51±2.06	5.42±0.30

3.2. Characterization of biofilms

Table 2 demonstrates the results concerning the characterization of the physicochemical and mechanical properties of CL-LBG+ and CL-LBG biofilms:

3.2.1. Organoleptic properties of films

The sensory properties of foods or beverages greatly influence the intake behaviors far beyond the mere setting off, stimulating or curtailing the consumption desire and enhancing the conveyed post-ingestive experiences (Forde, 2016). Overall, the predominant opinion of the panel juries on both films was very positive (Fig. 1). The smell of caramel discriminated both films. The CL-LBG film had a sour taste against a more pleasant sweet caramel taste for the CL-LBG+. Without polyphenols, the film was brown and smooth, containing scattered granules, with a continuous and uniform surface. The CL-LBG+ had a greenish brown color and smooth homogeneous and coherent appearance (Fig. 1). These results are similar to the results of Gheribi et al. (2019), unlike the biofilms of Lira-Vargas et al. (2014), having a lumpy structure with dispersed granules regardless of the variety of mucilage used, hence a discontinuous structure.

3.2.2. Film thickness and density

Thickness is an essential parameter to be considered, as changes in structural, thermal, mechanical or barrier properties should be expected in films with inhomogeneous thickness values (García et al., 2020). Likewise, film density has a direct impact on tensile strength, elongation and WVP (Li et al., 2020). The results mentioned in Table 2 show that CL-LBG+ (0.052±0.002 mm) were thicker than CL-LBG (0.030±0.0013 mm), which confirms a substantial positive influence of polyphenols on this parameter. The studied films are thicker than films prepared by Lira-Vargas et al. (2014) with 0.03 to 0.04 mm but thinner than those prepared by Gheribi et al. (2018) and Espino-Diaz et al. (2010) (0.180 ±0.006 mm and 0.184±0.005 mm). As interpreted by Espino-Diaz et al. (2010) and Gheribi et al. (2018), the thickness of biofilm varies depending on the type of ingredients used, the type of mucilage/gum and their composition, the amount of plasticizer and the method of manufacture. According to Lin et al. (2022), an increased concentration

Table 2
Physicochemical and mechanical properties of films.

Property	Films	
	CL-LBG+	CL-LBG
Thickness (mm)	0.052±0.002	0.030±0.0013
Density (g/mL)	0.11±0.01	0.51±0.004
Humidity(%)	33.74±0.91	29.41±0.58
Solubility (%)	71.21±1.26	94.99±1.33
Contact angle (°)	55.23±0.32	21.86±0.54
Water vapour permeability (WVP) ($g \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-1}$)	$2.97 \times 10^{-6} \pm 0.40 \times 10^{-6}$	$2.45 \times 10^{-8} \pm 0.29 \times 10^{-8}$
Light transmission and transparency (%)	29.74 ± 0.70	19.63 ± 0.48

(a) (b)

Fig. 1. Mucilage/Locust bean gum film (a) with Opuntia cladodes polyphenols CL-LBG+ and (b) without Opuntia cladodes polyphenols CL-LBG.

of polyphenols can cause greater covalent cross-linking between polymers, reducing the tendency to shrink during drying and giving a greater thickness. The density of CL-LBG+ was lower than CL-LBG (0.11±0.01. g/mL versus 0.51±0.004 g/mL). These results oppose the significant increase ($p < 0.05$) in density with the increase in the concentration of tea polyphenols noted in the study of Sun et al. (2017). An intercalation action of polyphenols on mucilage/LBG copolymer could be proposed to explain this effect. Accordingly, Sun et al. (2017) stated an intensification of interactions between polyphenols and polymers as the concentration of polyphenols augmented.

3.2.3. Moisture content

It should be mentioned that film water content is an important factor for evaluating their mechanical properties, seeing the plasticizer role of water. On the other hand, high water vapor permeability (WVP) values are due to higher water content in films (Chiou et al., 2009). In this study (Table 2), the moisture content of films fluctuates from 33.74±0.91 % for CL-LBG+ to 29.41±0.58 % for CL-LBG. These results oppose the results found by Lin et al. (2022). These authors showed an inversely proportional relation between the addition of apple polyphenols and the film humidity, which dropped from 17.14±2.15 to 12.47±0.84 %, and explained this effect by the reticulating action of the polyphenols on the dry materials forming the film. The existing differences among phenolic extracts and their added doses could be suggested as arguments for the contradiction of results. Moreover, the assessed films are more humid because of their particular composition in hygroscopic ingredients. The addition of galactomannans increases water-binding capacity but decreases it if these polysaccharides are added again (Arda et al., 2009). Mucilage can retain large amounts of water regardless of the nature of the growing site. In addition, the presence of glycerol as a plasticizer of hydrocolloid films increases their humidity. It is a highly hygroscopic plasticizer with several OH functions that easily retain water in the film matrix during the drying and storage process (Ghasemlou et al., 2011; Razavi et al., 2015; Zhang et al., 2016; Gheribi et al., 2018).

3.2.4. Solubility in water

The water solubility of edible films is an essential factor affecting their final application. In practice, high solubility is required for products needing easy dissolution before consumption, while low solubility adapts to products necessitating water resistance during treatment and storage (Ghasemlou et al., 2011). The film resistance increases with decreasing water solubility index (Lin et al., 2022). According to the results (Table 2), a greater hydro-solubility of CL-LBG was noticed when compared to active ones (94.99±1.33 % against 71.21±1.26 %). Therefore, the active films are more resistant to water than control films. This characteristic of films is dependent on the number of free OH groups available in polymeric matrix since they can form hydrogen bonds between the polymers (Araújo et al., 2018). Hence, the lower water solubility in active films observed in this work could be attributed to the powerful interaction between polyphenols and other compounds, inhibiting the contact between H_2O molecules and OH molecules (Lin et al., 2022).

3.2.5. Water contact angle

The contact angle with water is one of the fundamental properties of wetting of packaging films (Ma et al., 2017). The surface hydrophobicity of biofilms can be assessed by measuring the contact angle formed between the water droplet and the upper film face. The surface hydrophobic property increases parallelly to the increase in contact angle (Jouki et al., 2013). In this study, the contact angle of CL-LBG was less critical than CL-LBG+ (21.86±0.54° and 55.23±0.32°), which reflected a higher hydrophobicity of films in the presence of polyphenols (Table 2). Compared to the values of Gheribi et al. (2019), ranging from 84±3.25 to 86±3.28°, the CL-LBG and CL-LBG+ are more hydrophilic. Similarly, to these results, the polysaccharide-based edible films are partially wettable and usually had a water angle contact in the scope of 30° and 90° (Moussaoui et al., 2022). The copolymerization between polyphenols and reactive groups of constitutive polysaccharides may fulfill the free volume in the structural network, which enhances its hydrophobicity and, consequently, its water barrier properties (Jakobek, 2015).

3.2.6. Film water vapor permeability (WVP)

WVP is a crucial index for determining the mobility of water between food and the environment, as it is considered a critical metric predicting the barrier effect of edible film (Wang et al., 2021). Referred to the quantity of moisture passing through a unit area of material per unit time, the WVP is governed by the thickness and composition of films. Thus, low rates of WVP may reduce the moisture loss of products and extend their shelf life. This tool is easily exploited to predict changes in food water content (Nogueira et al., 2018; Vickers et al., 2022). The WVP of the CL-LBG films was lower than CL-LBG+ films ($2.45 \times 10^{-8} \pm 0.29 \times 10^{-8}$ against $2.97 \times 10^{-6} \pm 0.40 \times 10^{-6}$ $g \bullet m^{-1} \bullet s^{-1} \bullet Pa^{-1}$) (Table 2). The presence of those phenolic polymers accentuates the WVP, which lowered the film barrier effect in the face of water vapor. This increase is also reported by Lin et al. (2022). The hydroxyl-rich structure of polyphenols, and when the concentration of these exceeds a specific value, the excess of -OH interacts with H_2O molecules to make the membrane more hydrophilic. Excess polyphenols can reduce density between polymer arrays, increasing WVP to some extent (Wu et al., 2019). In addition, increasing the thickness of the film makes it more difficult to get water out and increases its pressure in the film, another reason for high WVP (Vickers et al., 2022).

3.2.7. Light transmission rate and transparency of films

Transparency stands as a critical attribute in the selection of packaging films suitable for food products. Certain photosensitive foods necessitate opaque films to withstand light exposure better, consequently extending their shelf life (Guzman-Puyol et al., 2022). CL-LBG+ exhibited higher transparency compared to CL-LBG, with values of 29.74 ± 0.70 % and 19.63 ± 0.48 %, respectively (Table 2). Both composite films demonstrated greater transparency than pure or crude mucilage/LBG films (7.90±0.75 % and 1.87±0.56 %, respectively), as reported by our previous work (Moussaoui et al., 2022). The crude (unpurified) mucilage and LBG were obtained through basic aqueous extraction and mechanical separation, while their purified forms were produced via solvent precipitation (ethanol for mucilage, isopropanol for LBG) (Moussaoui et al., 2022). It's noteworthy to point out the inversely proportional relationship between film thickness and its transparency, which might partly explain the huge gap in opacity between those films of the same nature. Despite the similarity of the basic matrix of these films (mucilage and LBG) in both studies, it is evident that existing differences in raw material, extraction methods and film-forming techniques in each one impact heavily the overall quality of the formed film, including transparency. This claim is confirmed by the results of González Sandoval et al. (2019) on three films synthesized based on mucilage of different Opuntia ficus-indica cultivars "Jalpa, Villanueva and Copena", getting respective rates of 7.431 ± 0.895, 4.674 ± 0.364 and 3.817 ± 0.288. In the current case, polyphenols increment made films, already more transparent by copolymerization of mucilage and LBG polysaccharides, even more transparent. Gorgieva and Kokol (2011) also indicated that the transparency of a polymer film is contingent upon the ingredients used and their interactions, which are founded on the development of hydrogen bonds. The heightened transparency of the films can be attributed to the porous and open structure of the copolymer network, suggesting a low tortuosity in light transmission (Lira-Vargas et al., 2014). The marked low opacity suggested a good adequacy of active films for foods that need a better visual appearance yet insensitive to light exposure.

3.2.8. ATR-FTIR

FTIR spectroscopy aimed to identify the distinctive functional groups present in the compounds and to assess the conjugation that occurred between the mucilage and locust bean gum polysaccharides, plasticizers and polyphenols. Noticeably, that all fabricated films had a quite similar spectrum with small differences due to the presence of polyphenol molecules (Fig. 2). This resemblance may be contingent on the particular structure "–OH groups rich hydrocarbons" of film manufacturing components, namely, mucilage, carob gum, glycerol and polyphenols (Hashemi Gahruie et al., 2020). Nevertheless, dissimilarities including the disappearance and coupling of peaks allude to the reactions or interactions of the said functional groups. These reactions mainly affect the barrier, mechanical and thermal properties of films (Gheribi et al., 2019). The broad spectrum peaks between 3000 and 3500 cm^{-1} in both films revealed the stretching vibrations of free –OH, hydrogen-bonded OH or glycerol -OH bond as stated by Davachi et al. (2021). Furthermore, it was associated with polyphenols or water's OH stretching vibration mode (García et al., 2020). This enormous peak can also be attributed to the OH groups' hydrogen bonding inside glucopyranose rings (Hashemi Gahruie et al., 2020). According to Gheribi et al. (2019), this large band of OH stretching is possibly related to alcohol and carboxylic acid involved in OH intermolecular bonding. This large absorbance peak could be more intense and shifted to lower frequencies following the hydrogen bonding between the polysaccharide matrix, plasticizers and/or polyphenols as a result of the additional OH groups provided by these added polyol structures (Gheribi et al., 2019). This finding was observed for glycerol-plasticized films, affirming that glycerol enhances the plasticity of carbohydrate-based films, rendering them more flexible (Gheribi et al., 2019). Overall, this band expressed inter/intramolecular links of OH groups of adjacent molecules forming the principle chains of matrix polysaccharides, besides the binding of H-bonds between them, glycerol and water molecules (Ayquipa-Cuellar et al., 2021). In addition, the absorption peaks around 2960 cm^{-1} and 2855 cm^{-1} are assigned to the typical asymmetric stretching vibration of C—H in aliphatic CH_3 or CH_2 groups, respectively (Gheribi et al., 2019; García et al., 2020; Davachi et al., 2021). The band of ~2900cm^{-1} corresponds to the stretching vibration of hydroxymethylene groups ubiquitous in the pyranose and furanose conformations of the most frequent monosaccharides of Opuntia mucilage; xylose, galactose, and arabinose (Quintero-García et al., 2021). Hence, a shoulder band was marked at ~1700 cm^{-1} only for CL-LBG and attributed to carbonyl group C=O stretching by many authors (Gheribi et al., 2019; Aragón-Gutiérrez et al., 2020; Valdés et al., 2021). The absorption peak observed at ~1600 cm^{-1} in both spectra is attributed to COO^- asymmetric and symmetric stretching, as noted in the research of Gheribi et al. (2019) and Quintero-García et al. (2021). In both films, the 1600 cm^{-1} band split into a second one of a lower frequency (~1400cm^{-1}), translated preponderantly the establishment of H bonds between carboxylic functions and other film constituents, a mechanism destabilized by the molecular structures of reactant

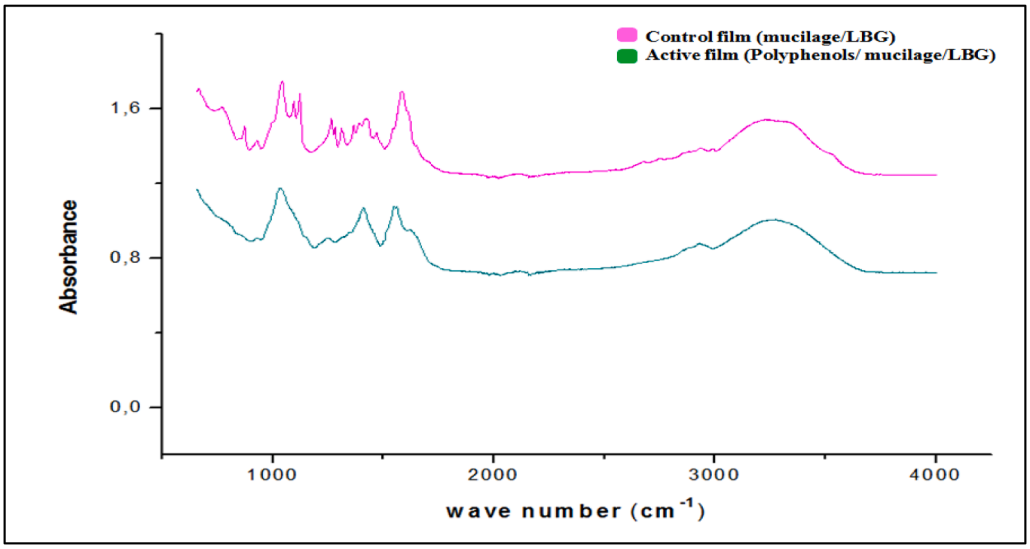


Fig. 2. ATR-FTIR spectrum of polyphenolic mucilage/LBG film (green line) and control mucilage/LBG film (pink line).

polyphenols providing abundantly non H-bond interactions. The result was a higher peak at 1400 cm^{-1} in free-polyphenol films. 1651 and 1417 cm^{-1} represent, respectively, the C–O–O asymmetric and symmetric stretching bands (Davachi et al., 2021). Moreover, the set of peaks between 1400 and 1200 cm^{-1} was assigned to C—H or O—H vibration similarly to the results of Gheribi et al. (2019) and Valdés et al. (2021). All samples show characteristic bands between 800 and 1200 cm^{-1} in the polysaccharide fingerprint region (Davachi et al., 2021). The peak around 1033 cm^{-1} denotes C—O-C glycosidic linkage in polyphenolic and non-polyphenolic plasticized films. On the other hand, the two peaks approximating or exceeding 1100cm^{-1} and existing only in the films enriched with polyphenols belong to the C—O stretching like in ester groups or even a C–OH deformation vibration. These indications were identically shown by García et al. (2020), Hashemi Gahruie et al. (2020), Davachi et al. (2021) and Valdés et al. (2021). Furthermore, a symmetric C–O–C stretching peak of the ester group is possibly identified at ~ 1160 cm^{-1}, which was also the case in the work of Valdés et al. (2021). The creation of interactions between functional groups of carbohydrate matrix and water, plasticizers and polyphenols during the film-forming process could be suggested to clarify these results. Gheribi et al. (2019) affirmed that molecular connections take place among the functional groups in the presence of water and plasticizers. The presence of polyphenols promotes this phenomenon and affords more available functional groups (OH, COOH…) to get potentially different types of linkages. Finally, the absorption range from 800 to 900 cm^{-1} was attributed to monosaccharides of locust bean gum; for instance, α-D-galactopyranose and β-D-mannopyranose were related to the absorption at 814 cm^{-1} and 873 cm^{-1} by Tako et al. (2018). In this array, these peaks are inexistent in non-polyphenolic film, reflecting the lack of this monomeric form in this film model subsequently to a more uniform and stable networking of the polysaccharides in absence of polyphenol units according to the previous statements.

3.2.9. NMR

The attached molecular structure established between polyphenols and polysaccharides can be characterized by ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy (Fig. 3). Therefore, NMR can uncover detailed information on polyphenol–polysaccharide conjugates, including the inserting positions between them and the degree of substitution (Guo et al., 2022). In this work, the narrow crowded region between 3 and 5 ppm is typical of polysaccharides and approves the presence of numerous comparable sugar residues (Singh & Bothara, 2015). Locust bean gum is a typical representative of galactomannans. It is a polysaccharide that contains a β-D mannan backbone where galactose is linked at the O-6 position of the mannosyl residue. Besides, mucilage is another type of plant polysaccharides consisting of various

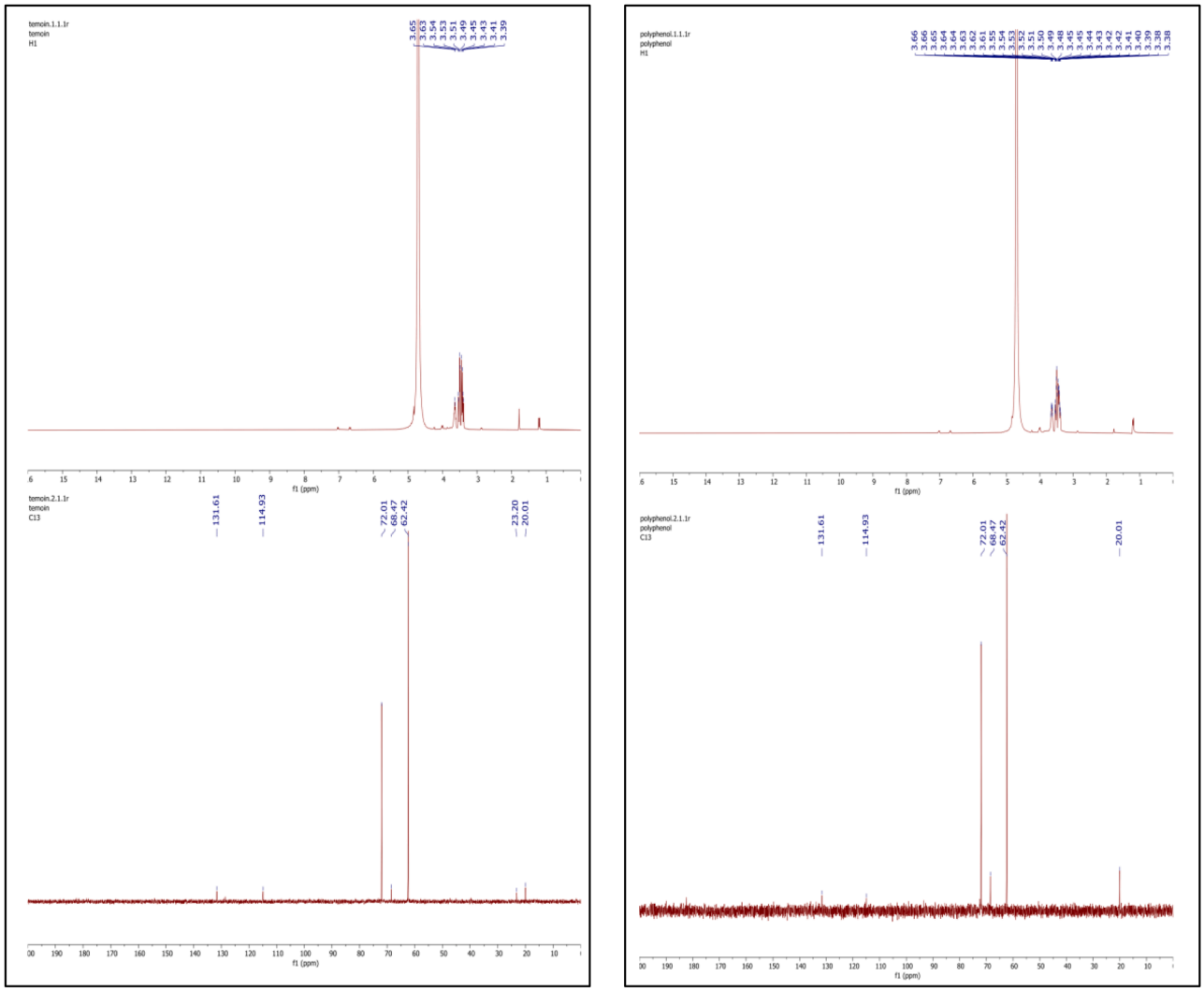


Fig. 3. ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectrum of (a) CL-LBG film and (b) CL-LBG+ film.

sugar units, such as arabinose, galactose, mannose, and xylose (Singh & Bothara, 2015). According to the ^{1}H NMR spectrum, the cluster of signals remarked between 3.5 and 3.64 ppm was also noted between 3.5 and 4.2 by Radziej et al. (2021, 2022) and corresponded to the protons of the locust bean gum sugar rings. Additionally, the ^{1}H NMR signals of mucilage sugar rings protons, except anomeric ones attached to the hemiacetal or hemiketal carbons, are commonly in the range of 3 to 4.5 ppm, and can be used to identify the type of sugar and its position in the polysaccharide chain (Singh & Bothara, 2015). The signals 3.60–3.65 ppm can be attributed to OH and CH groups of mucilage mannose, while peaks included between 3.55 and 3.81 ppm may be qualified as the CH_2 group of arabinose in the same polysaccharide (Singh & Bothara, 2015). The peak at 3.38 is probably associated with the CH group of C-5 of unesterified galacturonic acid, whereas the peak at 3.64 ppm along with that of ^{13}C NMR 72 ppm might be related to the rhamnose unit (Reyes-Ocampo et al., 2019). The 4.7 ppm signal might come from the anomeric H_1 of b-D-glucuronic acid units of mucilage (Zhao et al., 2007). Moreover, the presence of (1→4) linked β-D Galactose could be proven by the signal 3.64, as cited by Elshewy et al. (2023). Elshewy et al. (2023) divided the ^{13}C NMR spectrum of Opuntia mucilage into chemical shift regions: alkyl C (0–45 ppm), O-alkyl C (45–110 ppm), olefinic and aromatic C (110–160 ppm), phenolic C (140–160 ppm), and carbonyl C (160–220 ppm). The signal 20.01 ppm is possibly caused by the presence of CH_3 in unesterified rhamnose (Reyes-Ocampo et al., 2019). The signal at 23.20 ppm is identified as the carbonyl groups of N-acetylglucosamine by Guo et al. (2022). Meanwhile, the signal at 72 ppm was the highest peak and could be attached to the CH group of rhamnose or mannose of mucilage, as stated by Singh and Bothara (2015). The peaks of the carbons other than C=O groups or anomeric carbons were between 60.1 and 80.9 ppm (Singh & Bothara, 2015). Moreover, the signals noted at 114.92, 128.59, and 131.60 ppm may be assigned to aromatic and phenolic compounds according to Gheribi et al. (2019) and Elshewy et al. (2023). In NMR spectroscopy, a shift towards fewer peaks typically indicates an association or aggregation of constitutive molecules with each other or a complex formation with other molecules, leading to a reduction in the number of peaks. Furthermore, getting lower ppm values reflected a decreased electron density around the observed nucleus known as deshielding, subsequently to the phenomenon of conformational change and interaction with other molecules undergone by the analyzed molecule.

3.3. Film activities

Table 3 provides the polyphenol content of the filmogenic solution and the active film, and the antioxidant activity of the films and after solubilization in food simulants:

3.3.1. Total phenolic content (TPC) of the active film

The supplementation of the CL-LBG+ with polyphenols increased its content by 7.68 mg GAE/mL compared to the CL-LBG ($p < 0.05$) (Table 3). The drying temperature not exceeding 40 °C preserved the polyphenolic enrichment of active films well.

3.3.2. Antioxidant activity of films (DPPH inhibition)

This test was important to verify that the embedment of polyphenol molecules in the film matrix conferred antioxidant activity to the packaged foodstuff. The mechanism of action of this class of natural antioxidants is essentially founded on blocking the formation of reactive nitrogen or oxygen species and/or on scavenging free radicals. The resistance to oxidation of packaging materials has an attenuating effect on the rate of food oxidation and subsequently a significant impact on extending its shelf life (Lin et al., 2022). The antioxidant capacity of films with or without polyphenols, expressed as a percentage of DPPH scavenging, showed superiority of CL-LBG+ by 6.33 % ($p < 0.05$) (Table 3). A dose-dependent relationship between polyphenols and the antioxidant capacity of films can be proposed, aligning with the findings of Lin et al. (2022). It has been reported that the antioxidant efficacy of biodegradable films correlates with the amount of added antioxidant agents (Gómez-Estaca et al., 2009). In addition, numerous studies on natural products have demonstrated the responsibility of phenolic compounds, in particular for their antioxidant activities, since these biological molecules are known to possess antioxidant capabilities owing to their unique structure and chemical characteristics (Gómez-Estaca et al., 2009). The remarkable antioxidant capacity of polyphenols was directly related to the high number of structural-OH groups, owning an immense potential for hydrogen-atom transfer (Valdés et al., 2021). Likewise, several factors influence the antioxidant potential of polyphenols, counting reaction conditions like time, Antioxidant/DPPH ratio, solvent type and pH. Although, this feature is primarily linked to the phenolic profile of extracts. Moreover, Valdés et al. (2021) stated a concrete connection between phenolic agents' antioxidant behavior and biofilms structural particularity due to interactions between components in their matrix, which can modulate the migration of antioxidant compounds from the polymer matrix. From another standpoint, these researchers conveyed the repercussion of active constituents freeing from the biopolymer matrix by diffusion through the expanded structure of the entire antioxidant phenomenon. Similarly, the chemical crosslinking degree of films may be considered as a supplementary affecting factor able to control the rate of efficacious free phenolic antioxidants in the film network, consequently prompting the global hydrogen donor ability, thus the antioxidant activity (Valdés et al., 2021).

Table 3
Total Polyphenolic Content (TPC), and Antioxidant Activity (AA) in food simulants of films with or without polyphenols.

Film/solution	TPC (mg GAE/mL)	Antioxidant Activity (%)
Active film	27.40±0.11^{a*}	49.79±0.99^{a}
Control film	19.72±1.33^{b*}	43.46±0.16^{b}

Mean values with different superscript are significantly different ($p < 0.05$) when using ANOVA.
* Phenolic content in 1 ml is equivalent to the content in 0.2 cm^2 of film.

3.3.3. Antioxidant activity in food simulants

The antioxidant properties of the CL-LBG+ released in two food simulants, 10 % and 95 % ethanol (v/v), representing successively aqueous and fatty food, were also assessed through anti-radical activity over 24 h (Fig. 4). In the aqueous simulant, the antioxidant activity of CL-LBG+ increased steadily over the 24H up to a maximum of 60.55 ±2.03 % DPPH Inhibition. Regarding the lipidic simulant, the antioxidant activity of tested CL-LBG+ attained a threshold of 68.03±1.13 % after 24 h. From these results, a divide of 7.48 % was observed between

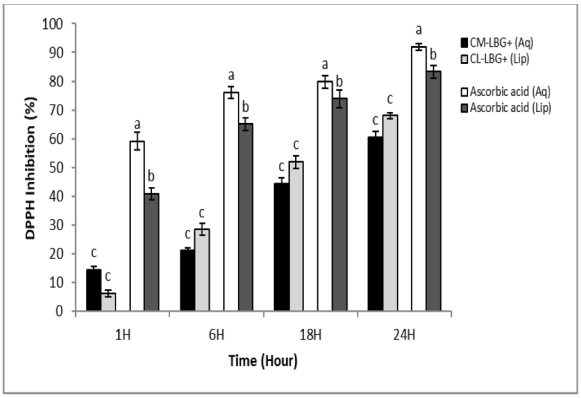


Fig. 4. Antioxidant activity of CL-LBG+ and ascorbic acid in aqueous and lipidic food simulants (Mean values with different superscript are significantly different ($p < 0.05$) when using ANOVA).

the antioxidant efficiency in aqueous and fatty simulants, reflecting the convenience of fat-rich food in getting a greater radical scavenging property of films ($p > 0.05$). A similar tendency was observed for ascorbic acid, permanently more effective than formed films in both cases, to give significantly ($p < 0.05$) higher rates peaking at 91.89±1.07 % in aqueous simulant, whereas it exhibited a value of 83.27±2.22 % ($p < 0.05$) at the same period in lipidic simulant. An identical conclusion was made by Aragón-Gutiérrez et al. (2020) on ethylene vinyl alcohol (EVOH) copolymer containing low amounts of phenolic compound, ferulic acid. Significantly greater antioxidant activity was reached in the 95 % ethanol simulant compared to the 10 % ethanol simulant, all in good accordance with the accentuated solubility of antioxidants alike in alcohols, and along with an enhancement of molecular mobility in the polymer triggered by a plasticizing action of ethanol, improving as a consequence the release of ferulic acid to the medium.

These findings align consistently with earlier studies investigating bioactive phenolic compounds, demonstrating notable antioxidant capabilities and exhibiting robust protective effects on food when liberated from the polymer matrix, as evidenced in previous literature (López de Dicastillo et al., 2011; Domínguez et al., 2018). It is proposed that the antioxidant activity is closely related to the levels of phenolic compounds released in each simulant, which is governed per se by the polarity of the solvent used, their degree of polymerization, as well as their interaction with matrix constituents. Due to their structural features, some phenolics dissolve better in organic solvents than in water, and their content, as well as the inherent antioxidation response, is therefore higher in the lipidic simulant (Naczk & Shahidi, 2004).

3.4. Application of mucilage/LBG active films as packaging coatings on peeled apples

In simple terms, shelf life is defined as “the time, under defined storage conditions, during which food remains safe, retains desired sensory, chemical, physical and biological characteristics as well as complies with any label declaration” (Manzocco et al., 2010). The impact of application of films on apple slices over 9 days of cold storage is presented in the Fig. 5.

3.4.1. Effect of coating on the weight of apples

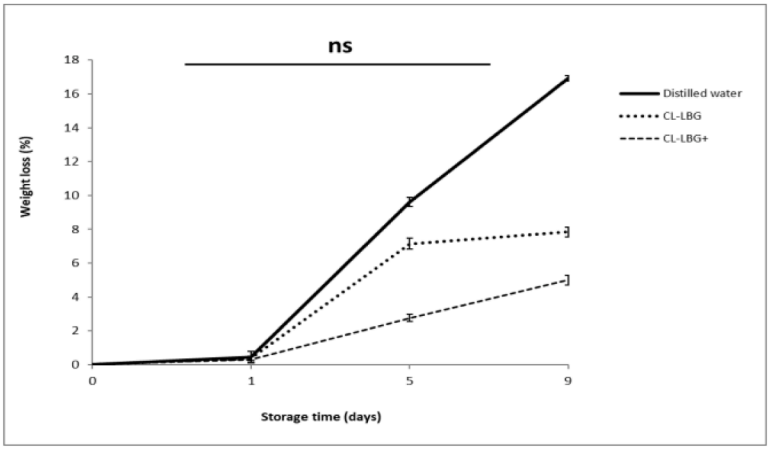
Fruits are prone to weight loss “water loss”, a key factor of postharvest deterioration leading to wilting, shriveling and browning, loss in firmness, flavor, and saleable weight, in addition to senescence acceleration (Lufu et al., 2020; Shinga et al., 2023). The fruit mass loss is a consequence of various processes, including respiration, transpiration, and other mechanisms through which ethylene gas, volatile organic compounds, and aromatic compounds may be lost (Bovi et al., 2018). The weight loss of the fruits increases with the storage time regardless of the treatment. However, from the 5th day onwards, the CL-LBG+ best retained the initial weight of apple slices, resulting not significantly ($p > 0.05$) in the lowest weight loss of 5.00±0.29 %, followed by CL-LBG 7.84 ±0.28 %. Uncoated apples had lost the most overall weight loss, noting a fall of 16.92±0.17 % in the fresh fruit weight. Likely, fig fruits covered using Opuntia ficus-indica mucilage edible coating had a dramatically 3-fold lesser weight loss than control ones (Allegra et al., 2017). The postharvest weight loss fluctuates greatly among fresh fruits and vegetables following a wide gamut of factors, for instance, environmental temperature and humidity, differences between species and cultivars of the same species, on top of fruit factors including its surface structure and area/volume ratio, its porosity (stomata and lenticels), and even the thickness and composition of the cuticle (Lufu et al., 2020). Basically, a water loss of 5 % to 10 % of the initial weight of fruits and vegetables makes them unmarketable as a consequence of global quality damage and breeds a concomitant shortening of the shelf life of fruits (Lufu et al., 2020). The investigated CL-LBG+ granted alleviation in this weight loss and delayed the associated fruit ageing mechanisms like respiration rate and ethylene production. The composite polymer coating formed a semi-permeable barrier over the apple slices, slowing dehydration and establishing a controlled atmosphere with restricted passage for specific gases (Allegra et al., 2016; Kumar et al., 2021; Shinga et al., 2023).

3.4.2. Effect of coating on the pH of apples

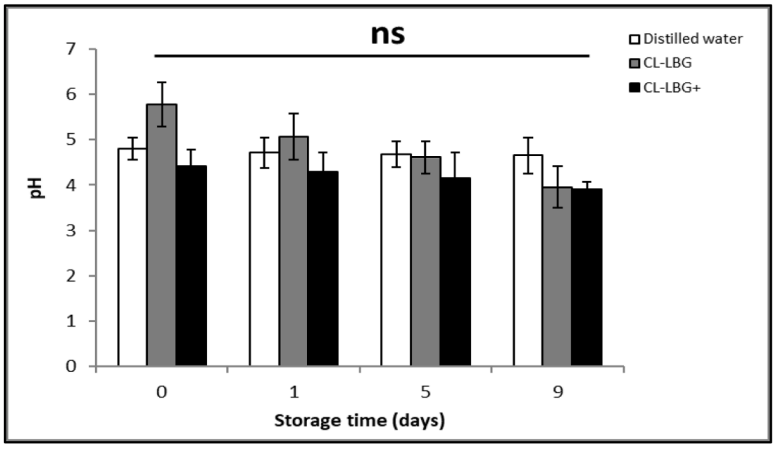
Changes in pH are quite indicative of fruits’ physiological changes and may affect food quality attributes, especially color. According to Fig. 5, fresh apple slices layered with CL-LBG+ were more acidic than CL-LBG apples and those immersed in distilled water (4.41±0.37, 5.78 ±0.49, and 4.80±0.25, respectively). In this optic, the acidic nature of coating solutions may be responsible for the primary low pH values of treated apple slices, as Rodríguez et al. (2020) mentioned. The pH of apples in all three groups experienced a decrease at an insignificant different extent ($p > 0.05$) after 9 days of storage at 4 °C, towards values of (3.91±0.16, 3.95±0.46, and 4.65±0.39) with CL-LBG+, CL-LBG, and distilled water, respectively. This pattern of pH-decrease in all samples over time, conducting towards significant differences at the end of the storage span, was also observed by Valdés et al. (2021). Above that, the superiority of pH values in the blank group versus treatments was equally cited by Rodríguez et al. (2020) for papaya edible films incorporated with Moringa oleifera and ascorbic acid on the 9th storage day. The pH of apples coated with distilled water was the least affected during cold storage, showing a mere regression of 0.15. The conversion of starch to sugar resulting from intensified respiration, along with the reduction of organic acids during maturation, increases the sugar/acid ratio and may therefore explain the stable pH of uncoated apples (Nandane et al., 2017; Abera et al., 2019). In the opposite, Nandane et al. (2017) confirmed that an effective coating would retain a low pH in fruit, which was the case in this study. It is possible to assume protection of organic acids from consumption and/or transformation of simple sugars in apple slices (Noshad et al., 2019). Coatings slow down metabolic reactions and respiration pace; thus, the senescence of fruits is delayed, and their shelf life is logically prolonged (Abera et al., 2019; Kumar et al., 2021; Moussaoui et al., 2022). Likewise, coatings were found effective in taking down the pH of fruit because of the deactivation of enzymes responsible for pH increase (Abera et al., 2019). Else, pH lowering can be very beneficial in deferring the early proliferation of spoilage microorganisms (Treviño-Garza et al., 2019).

3.4.3. Total soluble solids (TSS)

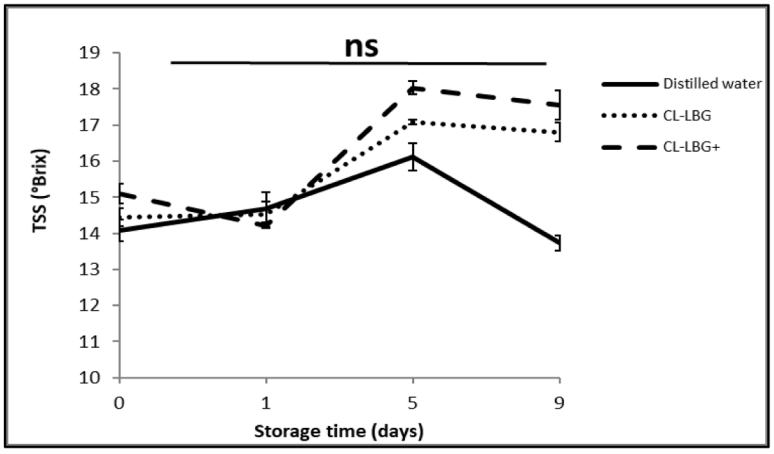
TSS is one of the primary drivers of consumer preferences for fruits (Shinga et al., 2023). Fig. 5 depicts the evolvement of TSS in coated and control apple slices. The initial value of TSS registered for distilled water apples, CL-LBG apples, and CL-LBG+ apples was respectively 14.08 ±0.31, 14.44±0.25 and 15.10± 0.28 °Brix. Clearly, the richness of the coating solution added superfluous soluble compounds able to heighten the native composition of the apples. Over time, TSS increases to touch on the 5th day a respective maximum of 16.12±0.38, 17.08±0.07, and 18.03±0.18 °Brix following the previous order of treatments, before declining to 13.74±0.21; 16.81±0.27 and 17.56±0.40 °Brix on the last day of storage. The difference between treatments was not significant ($p > 0.05$). A similar up-and-down fluctuation in TSS, culminating in the highest level for cactus mucilage-coated mango fruits after 16 days of storage compared to uncoated ones, was also reported by Abera et al. (2019). The initial elevation in TSS could be attributed to the transformation of starch into a readily usable form of carbohydrate. The subsequent decrease may also result from consuming sugar as a substrate for respiration and metabolic activities (Krishna & Rao, 2014; Abera et al., 2019). Ali et al. (2014) supposed an upregulation of TSS during fruit maturation versus a downregulation in ripe fruits attributable to respiration. Another possible explanation for the increase in TSS will be the observed water loss endured by stored fruits. The solubilization of structural cell wall hemicelluloses and polyuronides in ripe fruits may also contribute (Ali et al., 2014; Treviño-Garza et al., 2019).



(a)



(b)



(c)

Fig. 5. (a) Weight loss (b) pH evolution and (c) Total soluble solids (TSS) of apple slices coated with CL-LBG+, CL-LBG and distilled water during 9 days of cold storage (ns: non-significant difference ($p > 0.05$) when using ANOVA).

3.4.4. Sensory analysis

The hedonic scale is used in affective sensory food testing to quantify the degree of liking or disgust for a food product. In this regard, the hedonic scale is a balanced scale with nine categories, including one at the neutral center, representing changes in the perceived hedonic tone by sensory panelists (Lawless & Heymann, 2010; Rodríguez et al., 2020). Fig. 6 provides the sensory analysis radar for coated and non-coated apples before and after nine days of storage at 4 °C. At the commencement of the cold storing, the fruit slices of all treatments had a score above 7 minimally to get respective indifferent statistical ($p > 0.05$) values of 8.11, 8.20, and 8.09 for uncoated, CL-LBG and CL-LBG+ slices, expressing that panelists appreciated the fruits very much. After the nine days had elapsed, substantial differences were recorded in all tested qualities among the three groups of apples. Panelists preferred CL-LBG+ apples, followed by CL-LBG fruits, and then control fruits dipped in distilled water. The CL-LBG+ was the most effective, encompassing scores in the margin of "I like slightly". It best preserved texture (6.39), while CL-LBG was more effective in odor preservation (5.90). Apples dipped in distilled water were scored poorly across all qualities (3.92 for global acceptability). The same order was noted by Moussaoui et al. (2022) since the pure and crude films used as coatings on strawberries stored at 5 °C for 7 days were more efficient compared to control fruits ($p < 0.05$). Fruit color and its change are crucial components of quality and are often used as the most critical indicators prompting the end users' acceptability of these fruits (Ali et al., 2014; Shinga et al., 2023). Treviño-Garza et al. (2019) and Kumar et al. (2021) connected the color protection of stored fruits to the inhibition of oxidative or enzymatic browning in the presence of the coats, along with their slowing down of gas migration and respiration rate during the chilling storage. The coating of apples better preserved their texture firmness compared to the control sample. Similar results have been reported for Banana, chilies and cantaloupe fruits (Ali et al., 2014; Treviño-Garza et al., 2019; Shinga et al., 2023). The protective effect of mucilage and LBG coatings on fruit texture during storage was underlined by Moussaoui et al. (2022). The hygroscopic properties of the coating ingredients retain surface moisture and enhance internal firmness. Additionally, coatings may suppress the activity of texture-degrading enzymes such as cellulase (CX), polygalacturonase (PG), and pectin methyl esterase (PME), thereby maintaining a fresh appearance and reducing economic losses during storage (Moussaoui et al., 2022; Shinga et al., 2023). In line with the previously mentioned odor loss in apples, Liguori et al. (2021) similarly found that the odor values of fresh-cut cantaloupe pieces declined over storage time across all treatments, with coated slices significantly preferred over uncoated ones (scores of 4.30 vs. 2.70). The application of coatings on fruits helps preserve several sensory properties highly appreciated by consumers, such as taste and aroma. As for overall acceptance in the current work, the privilege of coated fruits was recognized by Liguori et al. (2021), assigning scores of 7 and 8, way greater than control samples scores of 6 and 4. Sensory appreciation aligns with the results of changes in TSS, pH and weight loss, where slices of apples with CL-LBG+ coating were chosen as the best product. The effects of the coating as a semi-permeable barrier against O_2, CO_2, moisture, and solute

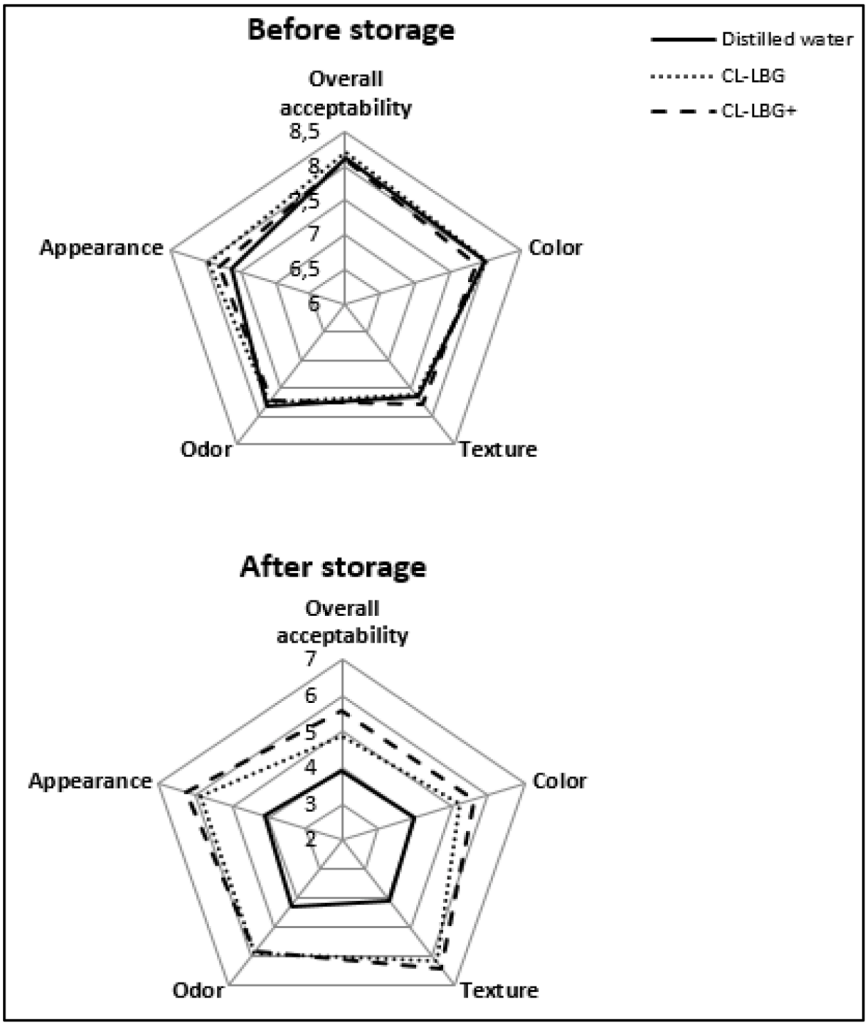


Fig. 6. Organoleptic analysis of apple slices coated with CL-LBG+, CL-LBG and distilled water before and after 9 days of cold storage.

mobilization may have contributed to the reduction of respiration cadence and oxidation rate (Ali et al., 2014). The intervention of CL-LBG+ coating in various aspects of organoleptic attributes, encircling color, odor, texture, and appearance, effectively preserved the desired overall quality of sliced and peeled apples, surpassing both marketability and edibility thresholds, therefore, prolonging subsequently their shelf life under cold conditions.

4. Conclusion

The CL-LBG+ film demonstrated a more appealing surface and aroma compared to CL-LBG. It was thicker and more impermeable to water vapor, less dense and hydro-soluble, along with a higher surface hydrophobicity and better transparency. FTIR and NMR analyses confirmed the presence of OH, COOH, and C=O functional groups, despite the intermolecular glycosidic and H bonds established through the network forming. The supplementation with phenolic extract led to a superior global antioxidant power in a dose-dependent pattern, as well as an increased antioxidant efficiency in fatty simulant compared to aqueous simulant. As a coating solution, the CL-LBG+ was the most effective in protecting apple slices' freshness, especially their texture. A controlling effect of active coatings on apple ripening process rythm and metabolic pace could be recognized, knowing their ef-fect on holding back the pH drop and TSS increase during cold warehousing. Looking ahead, over-coming the observed shortcomings of these films and refining their design under more conventional forms, such as plastic bags, remain very attractive options for positioning them as potential candidates for the food packaging industry.

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Informed consent statement

Informed consent was obtained from all volunteers involved in the sensory study according to the Helskin declarations.

CRediT authorship contribution statement

Badreddine Moussaoui: Conceptualization, Methodology, Validation, Writing – original draft, Writing – review & editing, Supervision. **Abdallah Rahali:** Conceptualization, Methodology. **Tahar Hanafi:** Writing – original draft, Visualization. **Laid Guemou:** Software, Data curation. **Kamel Zemour:** Data curation, Writing – review & editing. **Ali Riazi:** Supervision. **Othmane Merah:** Validation, Writing – review & editing, Supervision. **Sarra Halis:** Formal analysis, Investigation. **Ilham Hassouni:** Formal analysis, Investigation.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Merah reports was provided by University of Toulouse. If there are other authors, they 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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