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# Artichoke leaf extract coating on polylactic acid packaging to prolong fruit shelf life

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## ABSTRACT

The growing demand for safe and fresh food, coupled with the challenge of significant food loss and waste, has stimulated research on active packaging. In this study, artichoke leaf extract (ALE), obtained from food loss streams, was mixed with carboxymethyl cellulose (CMC) to develop an active coating on polylactic acid (PLA)-based packaging using a spray-coating approach. Prior surface modification of PLA using 1 % w/v PLA containing 20 wt% polyethylene glycol improved hydrophilicity and enhanced coating adhesion. Substituting ALE for CMC reduced coating solution viscosity, allowing a uniform coating. The inclusion of ALE conferred antioxidant (40–67 % DPPH scavenging) and antibacterial properties (2.11 and 1.08 log reduction against *E. coli* and *S. aureus*) to the coating, with controlled release governed by diffusion and swelling in lipophilic media (50 % ethanol) and predominantly swelling-controlled release in hydrophilic media (10 % ethanol and 3 % acetic acid). Analysis of the performance of active packaging in preservation of cut apples and strawberries demonstrated that ALE-coated PLA packaging effectively slowed oxidation, acid degradation, and microbial proliferation in cut apples and strawberries over 8 and 10 days storage, respectively, thereby improving overall freshness. While limited improvements were observed for firmness and browning in apples, coated packaging significantly enhanced phenolic content, antioxidant activity, and microbial stability, with strawberries showing the strongest protective effects. These findings highlight the potential of ALE-based coatings as a sustainable strategy to extend fruit shelf life and reduce food waste through active packaging solutions.

## 1. Introduction

In recent years, the importance of food preservation and safety in food production and processing has grown worldwide, largely due to population growth, resource depletion as well as increasing consumer demand for fresh and safe products [37]. Despite advances in food technology, a considerable amount of food is still lost due to quality degradation or spoilage during packaging and storage. At the same time, the extensive use of petroleum-based plastics in food packaging has generated serious environmental concerns due to their non-biodegradability and persistence in ecosystems [31]. To address these challenges, growing attention has been directed toward the development of environmentally friendly active food packaging systems. Such packaging is generally based on biodegradable polymer matrices incorporated with active agents that provide protective functions and extend food shelf life.

Among various biodegradable polymers, polylactic acid (PLA) has emerged as one of the most promising alternatives to petroleum-derived

plastics. PLA synthesized from renewable feedstocks such as corn starch or sugarcane, is industrially processable, biodegradable, and has a significantly lower carbon footprint compared to conventional plastics, while also offering good mechanical performance [7,23]. However, PLA alone lacks inherent antimicrobial and antioxidant activity, which are critical for prolonging shelf life and ensuring food safety. To overcome these limitations, PLA-based packaging can be functionalized with natural bioactive compounds that introduce antioxidant and antibacterial properties. Importantly, obtaining such bioactive agents from food loss and waste streams not only reduces waste but also adds environmental and economic value to active packaging innovations [38].

For instance, artichoke (*Cynara scolymus* L.) production generates a substantial amount of by-products, with approximately 70–80 % of the plant including leaves, stems, and roots discarded during harvesting and preparation. Global artichoke production reaches nearly 1978 kton annually, making these residues a significant source of underutilized biomass [4]. Notably, artichoke leaves are particularly rich in bioactive compounds such as polysaccharides and phenolics, which possess strong

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antioxidants and antimicrobial properties and therefore hold great potential for functional packaging applications [33].

Active packaging can be developed either by incorporating bioactive agents directly into the polymer matrix or by applying them as surface coatings. Coating methods are particularly advantageous for heat-sensitive natural compounds, as they help preserve functional activity, maintain the structural integrity of the packaging material, and ensure effective food contact [2,21]. Among a wide range of techniques such as electrospraying, dipping, cast coating, chemical vapor deposition, physical vapor deposition, roll-to-roll, and screen printing [49], spray coating is widely recognized as a suitable approach for industrial-scale applications. It is commonly used in production to apply coatings on large substrates, including plastics, metals, optical films, and functional surfaces, using both suspension and solution sprays [8]. For example, natural plant polyphenols and layered clays have been integrated into poly (vinyl alcohol)-based coatings on corona-treated PLA films, significantly enhancing active functions and gas barrier properties [51]. Similarly, cellulose nanocrystals modified with methacrylamide, cetyltrimethylammonium bromide, or zinc oxide have been spray-coated onto PLA films, resulting in strong antibacterial activity against *S. aureus* and *E. coli*, improved mechanical strength, reduced gas permeability, accelerated composability, and extended pork shelf life [20].

Significant progress has been made in developing active coatings on fruits and vegetables using various biopolymeric systems such as chitosan/quaternary ammonium salt/tannic acid [17], crosslinked chitosan/cellulose nanofiber- integrated with  $\gamma$ -cyclodextrin/curcumin [55], and hyaluronic acid integrated with cinnamaldehyde/hydroxypropyl- $\beta$ -cyclodextrin [54]. Despite these advancements, limited research has focused on applying natural bioactive compound-based coatings onto packaging materials like PLA for fruit preservation. Therefore, the present study aims to develop a bioactive coating for PLA-based food containers intended for direct food contact applications, where the active functionality is achieved through controlled migration and interaction at the food-coating interface. The coating formulation comprises artichoke leaf extract (ALE), a valorized food loss stream obtained through a green extraction approach, in combination with carboxymethyl cellulose (CMC), applied using a spray coating technique. Specifically, this study investigates strategies to enhance the surface compatibility of PLA with the coating solution, evaluates the functional and physical properties of the coated packaging, and assesses its effectiveness in preserving the quality of fresh fruits.

## 2. Materials and methods

### 2.1. Materials

PLA-based clamshells ( $11 \times 11 \text{ cm}^2$ ) containing aliphatic aromatic copolyester (PBAT) were kindly provided by Gaia Biomaterials (Sweden), which was prepared by thermoforming process. Artichoke leaves extract (ALE) was obtained using subcritical water extraction technique at  $150^\circ\text{C}$  and biomass to water ratio of 1:10 (g:mL) at two cycles of 20 min (1500 psi) as described in our previous work [33]. The freeze-dried fraction obtained from second extraction cycle containing more phenolics and lower carbohydrates compared to the first fraction was used in this study. ALE contained 20.94 % dry weight (DW) carbohydrate, 7.93 % DW starch, 4.35 % DW soluble protein, and 5.11 % DW total phenolic content was utilized in this research [33]. Other materials included PLA purchased from 123-Print (Jordbro, SE), and poly(ethylene glycol) (PEG, Mw  $\sim 8000$ ), glycerol, carboxymethyl cellulose sodium salt (CMC, medium viscosity), and chloroform obtained from Sigma-Aldrich.

### 2.2. Preparation of active coating

The surface of the PLA-based clamshells was modified prior to applying active coating in order to improve surface activity and polarity.

For this purpose, PLA solutions (1 % or 2 % w/v in chloroform) with various concentrations of PEG (0, 5, 10, 15, and 20 wt% of PLA) were prepared and sprayed ( $\sim 33 \mu\text{L}/\text{cm}^2$ ) onto the inner surface of the clamshells using an airbrush connected to a vacuum air pump, at a distance of 15 cm and a pressure of 2 bar. This experiment was conducted to determine the optimum formulation for enhancing surface hydrophilicity. Then, the solutions of CMC and ALE were prepared in distilled water at concentration of 2 % w/v separately and mixed at different ratios of 100:00, 70:30, 60:40, 50:50 (CMC: ALE). Then glycerol was added to the mixed solutions at 30 wt% and stirred for 30 min at room temperature. After preparing the coating solutions, they were sprayed ( $\sim 41 \mu\text{L}/\text{cm}^2$ ) onto the previously modified clamshells three times, allowing them to dry between spraying for better coverage and adhesion. The spray coating was done at 2.5 bar pressure and 15 cm distance from nozzle to the inner surface of clamshells. Different layers of active PLA clamshells are shown in Fig. 1.

### 2.3. Characterization of active packaging

#### 2.3.1. Contact angle

Contact angle measurements were performed using a tensiometer (Attension Theta Optical Tensiometer, Sweden) under ambient conditions to evaluate the effect of surface modification on the hydrophilicity of PLA-based clamshells. A  $13 \mu\text{L}$  droplet of deionized water was dispensed onto the modified clamshell surface using a micro syringe, and the angle formed between the droplet boundary and the surface was recorded after 10 s with OneAttension software (Biolin Scientific, Sweden). The mean contact angle was calculated from three replicate measurements.

#### 2.3.2. Hygroscopicity and water vapor permeability (WVP)

The hygroscopicity of the uncoated and coated samples was assessed to determine the coating's ability to absorb moisture from packaged food. Packaging samples ( $3 \times 3 \text{ cm}^2$ ) were placed in a high humidity chamber (100 % RH) and weighed every two days over a period of 10 days. Hygroscopicity was calculated from the weight change at each time point relative to the initial weight.

WVP of the PLA-based packaging was determined according to ASTM standard E96/96 M-24a. The samples were cut and mounted onto permeation cells (3.57 cm diameter; TQC Sheen, Zuid-Holland, NL) containing distilled water (RH = 100 %). The cells were sealed with caps and stored in a desiccator filled with silica gel at  $20^\circ\text{C}$ . Their weight was recorded periodically for one week, and WVP was calculated using Eq. (1).

$$\text{WVP} = \frac{\left(\frac{\text{g}}{\text{s}}\right) \times x}{A \Delta P} \quad (1)$$

Where g/t is the mass change (g) over time (s), x is the film thickness (m), A is the exposure area ( $\text{m}^2$ ), and  $\Delta P$  is the difference between vapor pressure of water and the silica environment (Pa).

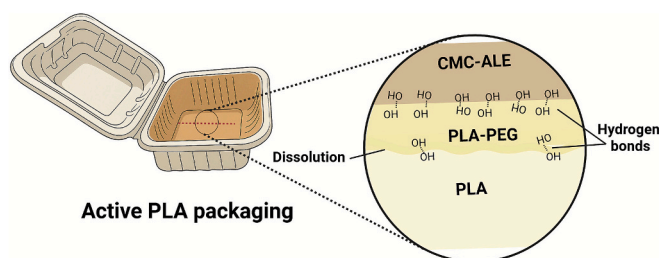


Fig. 1. Schematic representation of the different coating layers on the body surface of PLA-based packaging.

### 2.3.3. Fourier transformed infrared spectroscopy (FTIR)

Molecular interactions of the packaging before and after coating were analyzed using FTIR (Spectrum 3, Perkin Elmer, Waltham, USA) equipped with a Universal Attenuated Total Reflectance (ATR) device (GladiATR, Pike Technologies, Madison, USA) with a diamond crystal. Spectra were recorded in the range of 4000–400  $\text{cm}^{-1}$ , using 64 scans at a resolution of 4  $\text{cm}^{-1}$ .

### 2.3.4. Scanning Electron microscopy (SEM)

The surface microstructure and the morphology of the cross-section of the PLA-based clamshells before and after coating was observed by a benchtop SEM using a microscopy Phenom ProX (Phenom-World BV, Eindhoven, NL) operating at 10 kV of acceleration voltage.

### 2.3.5. Viscosity measurements of coating solution

The viscosity of the coating solutions was measured using a rheometer (Discovery HR-3, TA Instruments, USA) equipped with a Peltier plate–plate system. A smooth parallel plate geometry with a 40 mm diameter was used, and the coating solutions were loaded between the plates with a 1 mm gap. Measurements were performed at 25 °C over a shear rate range of 0.1–100  $\text{s}^{-1}$ .

### 2.3.6. Antioxidant activity

The antioxidant activity of packagings was measured by DPPH (1,1-diphenyl-2-picrylhydrazyl) scavenging assay. The experiment was performed in three different cycles of 15, 30, and 45 min by adding extra DPPH free radical in each cycle to determine how antioxidant activity of active packaging can be maintained over time. For this purpose, the specimen of control and different coated packaging (100 mg) was dissolved in 1 mL distilled water and then 0.5 mL of 0.2 mM methanolic solution of DPPH was added to the samples. The samples were incubated in dark for 45 min and 200  $\mu\text{L}$  of mixed solution were taken for analysis in intervals of 15 min and replaced with 200  $\mu\text{L}$  of new DPPH methanolic solution. The absorption of samples was recorded using a microplate reader (FLUOstar, BMG Labtech, Germany) at 517 nm and the remaining DPPH of each sample was calculated using the standard curve of DPPH to obtain radical scavenging activity (RSA%) of packaging.

### 2.3.7. Antibacterial activity

The antimicrobial activity of the coated PLA-based packaging was assessed against *Escherichia coli* (*E. coli*, CCUG 10979), and *Staphylococcus aureus* (*S. aureus*, CCUG 10778) as Gram-positive and Gram-negative representatives, respectively through colony counting method. The bacteria inoculum was prepared using Luria broth (LB) by incubation of their colony at 37 °C for 24 h and their optical density at 600 nm were adjusted to McFarland 0.5 standard ( $\sim 10^8 \text{ CFU.mL}^{-1}$ ). The packaging samples ( $1 \times 2 \text{ cm}^2$ ) were UV-sterilized on both surfaces and placed in sterile tubes containing 5 mL of Mueller Hinton broth inoculated with  $10^5 \text{ CFU.mL}^{-1}$  culture. The tubes were incubated at 37 °C for 24 h and afterward an aliquot of 100  $\mu\text{L}$  of the cultures were plated on Mueller Hinton Agar plates. The number of colonies was counted after 18 h of incubation at 37 °C and the bacterial growth inhibition was calculated in comparison with Mueller Hinton broth without any samples inoculated with bacteria as positive control.

### 2.3.8. Release of phenolics

The release kinetics of polyphenolic compounds in different food simulants were investigated according to Karim et al. and Li et al. [25,28] with some modification. For this purpose, different specimen of coatings ( $2.5 \times 2.5 \text{ cm}^2$ ) was immersed in 25 mL of 10 and 50 % v/v ethanol and 3 % v/v of acetic acid. The samples were shaken at 100 rpm at 4 °C to simulate the cold storage condition. Equal amounts of supernatant (500  $\mu\text{L}$ ) were taken at different times of independent experiments, and the polyphenol content was determined by the Folin-Ciocalteu method [6]. The release of polyphenols as a function of time was measured and normalized to the theoretical content. The Weibull,

first-order, Higuchi, and power law functions according to Eqs. (2)–(5) were used to investigate release kinetics. The model with the highest  $R^2$  value was considered to provide the best fit.

$$\frac{M_t}{M_\infty} = 1 - \exp\left[-(t/a)^b\right] \text{ Weibull} \quad (2)$$

$$\frac{M_t}{M_\infty} = 1 - \exp(-kt) \text{ First - Order} \quad (3)$$

$$\frac{M_t}{M_\infty} = Kt^{0.5} \text{ Higuchi} \quad (4)$$

$$\frac{M_t}{M_\infty} = K t^n \text{ Korsmeyer-Peppas (Power Law)} \quad (5)$$

Where  $M_t$  is the released amount of active compound at time  $t$ ,  $M_\infty$  is the released amount of active compound at equilibrium, and  $a$ ,  $b$ , and  $K$  are kinetic constants and  $n$  is the diffusional exponent.

## 2.4. Application of active packaging in shelf-life study of fruits

Active clamshells with the best balance of bioactivity, moisture absorption, and coating flowability were used to evaluate the shelf life of cut apples and strawberries. Fresh fruits were obtained from local markets. For apples, the fruits were washed with distilled water, dried, and cut into  $\sim 2 \text{ cm}^3$  cubes using a sterilized cutter, then packed under aseptic conditions. For strawberries, fruits of uniform size and free from defects were selected. Four strawberries were packed per clamshell. The samples were divided into three groups of without packaging, packaged in uncoated clamshell and packaged in coated clamshells. Apple cut and strawberries samples were stored at 4 °C and 85 % RH for 8 and 10 days, respectively. Physicochemical and microbial analysis were performed at 2-day intervals for three replicates (see Supplementary data).

## 2.5. Statistical analysis

One-way Analysis of Variance (ANOVA) was used to assess statistical significance at a 5 % level. Significant differences among group means were further evaluated using Fisher's LSD post-hoc test. All statistical analyses were conducted using OriginPro 2023 Statistical Software (OriginLab Corporation, USA).

## 3. Results and discussion

### 3.1. Developing of active coating for PLA-based packaging

The hydrophobic character of PLA surface was a major challenge for the uniform spreading of aqueous active coatings. To address this limitation, the surface of PLA-based containers was modified using PLA solutions blended with different concentrations of PEG. This modification enhanced surface hydrophilicity by decreasing the contact angle value (Fig. S1) and promoted better interaction between the container surface and the active coating layer. In agreement with this finding, Sundar et al. [46] reported that blending PLA with 15 % PEG improved the hydrophilicity of PLA-coated paper, increasing its moisture content from 5.68 to 5.94 %. In the present study, two different concentrations of PLA (1 and 2 % w/v) were evaluated for surface modification. While no significant difference in contact angle values was observed, the higher concentration (2 % w/v) led to agglomeration of PLA-PEG on the surface, therefore, 1 % w/v PLA solution was selected for further experiments. To reduce the risks associated with food-contact materials, green solvents such as dimethyl carbonate, ethyl acetate or ethyl formate or their mixture can be considered safer alternatives to chloroform [29]. Various PEG concentrations (5–20 wt% of PLA) led to different effects on surface hydrophilicity (Fig. S1). By increasing the PEG concentration by 10 %, surface hydrophilicity unexpectedly started



decreasing, while higher concentrations enhanced hydrophilicity. A similar irregular trend was reported by Serra et al. [41] in PLA-PEG blends for 3D-printed scaffolds, where the addition of 20 % PEG also resulted in fluctuations in contact angle values. This behavior may be explained by initial hydrogen-bonding interactions between PLA and PEG in the modifying layer, which consume polar groups that would otherwise interact with water molecules. At higher PEG concentrations, PLA becomes saturated, and more polar groups of PEG are exposed to the surface, leading to increased hydrophilicity. Based on these observations, the formulation consisting of 1 % w/v PLA solution containing 20 wt% PEG was identified as the most suitable for surface modification of PLA-based containers.

The viscosity of active coating solutions plays a crucial role in the design of active packaging. Appropriate viscosity is essential to achieve a uniform distribution and to form a continuous and consistent active layer on the packaging surface. The CMC control solution (2 % w/v) exhibited significantly higher viscosity compared with those containing ALE, and it showed a shear-thinning behavior with increasing shear rate (Fig. S2). This property is generally beneficial during spray coating, since the solution becomes less viscous under shear stress. However, the viscosity reduction was insufficient to allow uniform spreading and coating of PLA films, as also confirmed by SEM observations (Fig. 1c). Incorporating ALE extracts into the CMC matrix markedly altered the rheological properties. Increasing the substitution level of ALE progressively reduced viscosity, and the flow behavior of the CMC solution became less dependent on shear rate. The greater stability of coating solutions at higher shear rates may be attributed to interactions between polyphenols and polysaccharides in ALE and CMC, where CMC acts as a cross-linking agent and stabilizes the system [10]. Comparable effects were also reported for alginate-based coatings containing olive leaf extract [32].

### 3.2. Microstructure of coated PLA-based packaging

The active coating solution formed a uniform and even layer on the PLA-based packaging surface. Surface modification with PLA-PEG and coating with plain CMC did not noticeably alter the appearance of the packaging. However, the inclusion of ALE in the coating solutions affected the color and visual appearance of the coated packaging (Fig. S3).

The surface microstructure and cross-section of the uncoated and different coated packaging are presented in Fig. 2. The surface of the neat PLA sample exhibited a rough texture with some particles (Fig. 2a). Surface modification with PLA-PEG smoothed the surface, creating a

continuous layer over the entire packaging (Fig. 2b). In contrast, coating with CMC alone was unable to fully cover the surface (Fig. 2c), likely due to its higher viscosity and lower flowability. Substituting 30 % of CMC with ALE resulted in a uniform and smooth coating, covering the surface completely without pores or cracks (Fig. 2d). However, further increasing the ALE content led to the appearance of particles and an increase in surface roughness (Fig. 2e and f).

Cross-sectional SEM images of neat PLA revealed a homogeneous and semi-porous structure (Fig. 2g). After surface modification, partial dissolution of the PLA surface was observed (Fig. 2h). The plain CMC coating showed minor gaps at the interface with the substrate (Fig. 2i), whereas the active coating (approximately 5  $\mu\text{m}$  thick) exhibited good adhesion to the pre-modified layer (Fig. 2j). Increasing the concentration of ALE in the coating further improved adhesion (Fig. 2k and l), likely due to the higher content of functional groups in ALE and the reduced viscosity of the coating solution, both of which enhance interfacial interactions between the layers.

The chemical structure and molecular interactions of the coating components were analyzed by FTIR (Fig. 3). In the spectra of plain and modified PLA samples, peaks at  $2840\text{--}2950\text{ cm}^{-1}$  corresponded to C-H

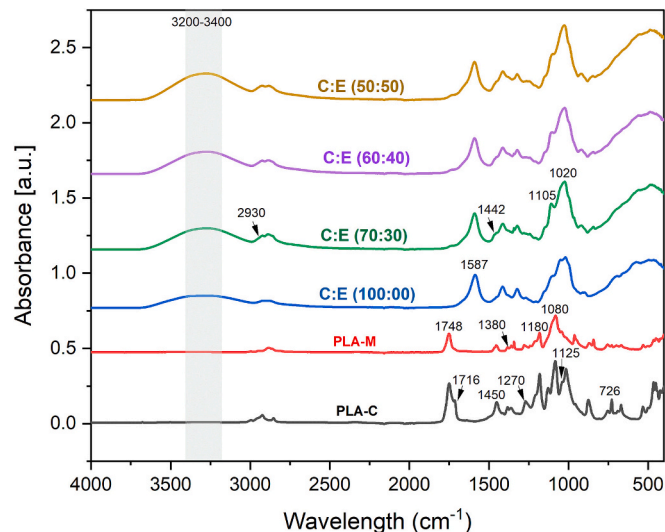


Fig. 3. FTIR spectra of PLA-based packaging including plain, surface-modified, and coated with carboxymethyl cellulose (CMC) and artichoke leaf extract (ALE).

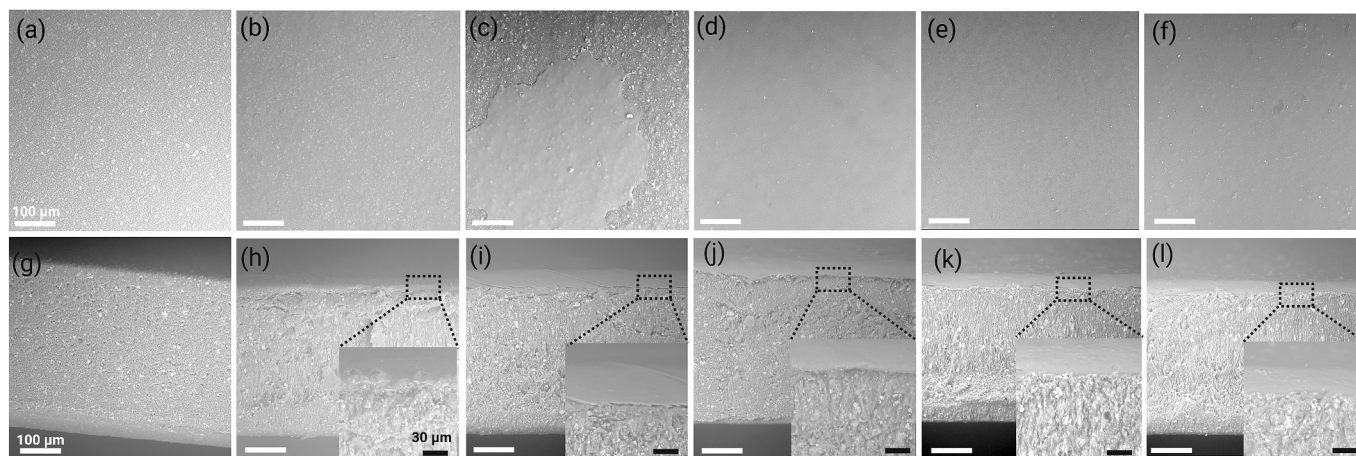


Fig. 2. SEM images of surface morphology (top, magnification  $\times 500$ ) and cross-sectional microstructure (bottom, magnifications  $\times 500$  and  $\times 2000$ ) of PLA-based packaging: (a, g) plain PLA; (b, h) surface-modified PLA; (c, i) coated with CMC:ALE (100:0); (d, j) coated with CMC:ALE (70:30); (e, k) coated with CMC:ALE (60:40); (f, l) coated with CMC:ALE (50:50).

stretching vibrations, while bands at 1748, 1080–1180, and 1450/1380  $\text{cm}^{-1}$  were attributed to C=O stretching, C–O–C stretching, and asymmetric/symmetric –CH bending, respectively [39,44]. On modified surface, weaker PLA bands and a shift of the 1748  $\text{cm}^{-1}$  peak indicated hydrogen bonding between C=O groups of PLA and –OH groups of PEG [44,46]. In contrast, plain PLA packaging showed additional bands from PBAT at 1716  $\text{cm}^{-1}$  (C=O stretching), 1270 and 1125  $\text{cm}^{-1}$  (aromatic/aliphatic C–O ester vibrations), and 726  $\text{cm}^{-1}$  ( $\text{CH}_2$  stretching) [50]. The plain CMC coating displayed a broad O–H stretching band at 3200–3400  $\text{cm}^{-1}$ , a 2900  $\text{cm}^{-1}$  peak from methylene C–H stretching, and characteristic peaks at 1587 and 1442  $\text{cm}^{-1}$  (asymmetric/symmetric –COOH stretching) as well as 1105 and 1020  $\text{cm}^{-1}$  (C–O–C stretching of the polysaccharide backbone). Incorporation of ALE caused slight changes in functional groups. It intensified absorption in the 3000–3500  $\text{cm}^{-1}$  region due to hydrogen bonding arising from O–H groups in phenolic compounds and polysaccharides (e.g., hemicellulose), along with enhanced bands in the 1000–1100  $\text{cm}^{-1}$  region linked to glycosidic vibrations and O–H bending [3,33]. The band near 2930  $\text{cm}^{-1}$  corresponds to O–H stretching of phenolic compounds in the active coatings [12]. Higher ALE concentrations increased the intensity of these regions, reflecting greater polysaccharide and phenolic content.

### 3.3. Moisture sensitivity of active packaging

The effect of different coatings on the WVP of PLA-based packaging is shown in Fig. 4a. Neat PLA exhibited the highest WVP, likely due to the high porosity observed in the cross-sectional SEM images. Surface modification with PLA–PEG significantly reduced WVP by 64 %, as the PLA–PEG layer effectively covered surface defects and created a more continuous, impermeable barrier to water vapor. The addition of plain CMC caused a slight, insignificant increase in WVP due to its hydrophilic nature, which promotes moisture absorption and retention. However, incorporating ALE into the coating resulted in a gradual decrease in WVP, possibly due to cross-linking interactions between ALE components and CMC that reduce the accessibility of polar groups. Nonetheless, these changes were not statistically significant.

The moisture absorption behavior of the uncoated and coated PLA samples under 100 % RH over 10 days is shown in Fig. 4b. All samples exhibited an increase in moisture uptake over time. The uncoated PLA absorbed the least moisture, reaching only 1.75 % after 10 days, reflecting its hydrophobic nature and the lack of polar functional groups for water interaction. Moisture uptake in neat PLA initially followed a linear trend, indicating gradual penetration, but eventually plateaued as the PLA matrix approached equilibrium with the surrounding humidity. In contrast, the active coated samples absorbed and retained

significantly more moisture, consistent with the hydrophilic character of CMC and ALE, as indicated by FTIR results. Increasing the ALE content in the coating further enhanced moisture absorption, likely due to the combined effects of higher hydrophilicity and structural characteristics of the coating matrix.

### 3.4. Antioxidant and antibacterial activity

Antioxidant activity is a key property of active packaging, enabling the preservation of proteins and lipids by preventing degradation and oxidation, thereby maintaining food quality and organoleptic properties. The antioxidant activity of the coated packaging was evaluated over three cycles by introducing additional DPPH free radicals at each stage. As shown in Fig. 5a, after 15 min of incubation, only the samples coated with CMC–ALE exhibited DPPH scavenging activity of approximately 40–44 %, compared to plain PLA and only CMC coated samples. Upon increasing incubation time to 30 min and DPPH concentration, the plain PLA showed scavenging activity around 17 %. This might be attributed to the release of packaging ingredients such as PBAT and plasticizers, which have known antioxidant properties [1,13,18,22], as well as the electron-donating ability of carboxyl groups in PLA chains [19]. Although the only CMC coated sample still didn't show any activity after 30 min, coatings containing ALE showed significantly highest antioxidant activity even with increasing DPPH concentrations. Higher ALE content led to greater activity, with scavenging rates of 54, 60, and 67 % for formulations containing 30, 40, and 50 % ALE, respectively. This enhancement is likely due to the sustained release of phenolic compounds over time. Even after 45 min in the presence of elevated DPPH levels, the coatings retained considerable activity (40–51 %), while plain PLA and only CMC coating showed the lower and no antioxidant activity, respectively. The antioxidant potential of ALE has been linked to phenolic acids and flavonoids capable of donating hydrogen atoms or electrons to neutralize reactive oxygen species (ROS) and free radicals generated during fruit tissue injury or respiration [24,48]. Phenolic compounds can also inhibit enzymes responsible for fruit browning, such as polyphenol oxidase (PPO), by acting as substrate analogs that bind to the enzyme's active site primarily through electrostatic interactions and hydrogen bonding [45].

The antibacterial activity of the active coatings against *E. coli* and *S. aureus* is presented in Fig. 5b. Plain PLA and only CMC coated samples showed slight but no significant bacterial reduction. In contrast, active coatings demonstrated a concentration-dependent reduction of *E. coli*, with 0.9, 1.96, and 2.11 log reductions for coatings containing 30, 40, and 50 % ALE, respectively. Against *S. aureus*, all active coatings achieved approximately 1 log reduction (0.91–1.08), with insignificant

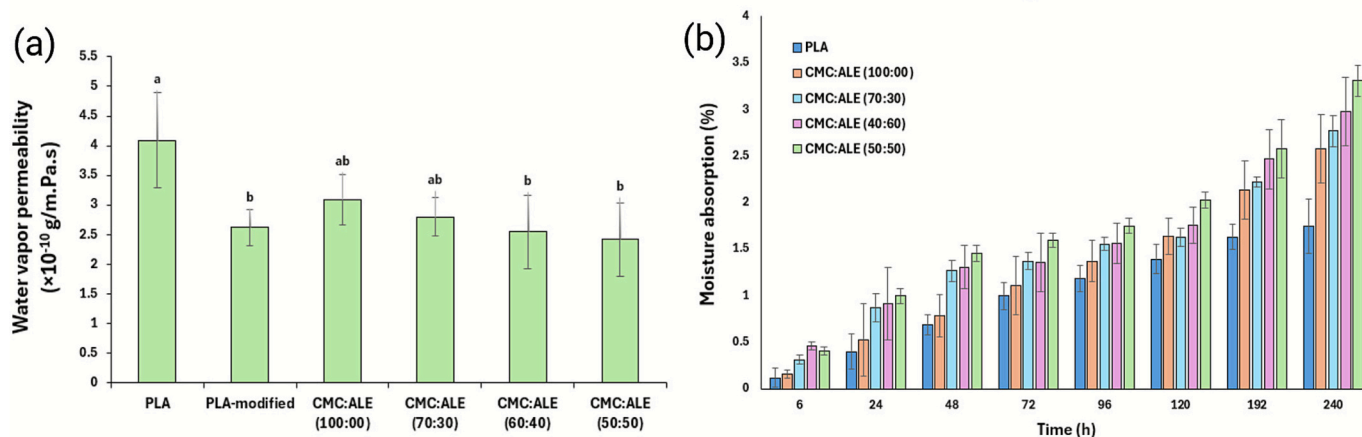


Fig. 4. (a) Water vapor permeability of plain, surface-modified PLA and PLA coated with different ratios of carboxymethyl cellulose (CMC) and artichoke leaf extract (ALE); (b) moisture absorption (%) of the same samples under 100 % relative humidity over 10 days (Different letters indicate significant differences among mean values of WVP across different groups ( $P < 0.05$ )).

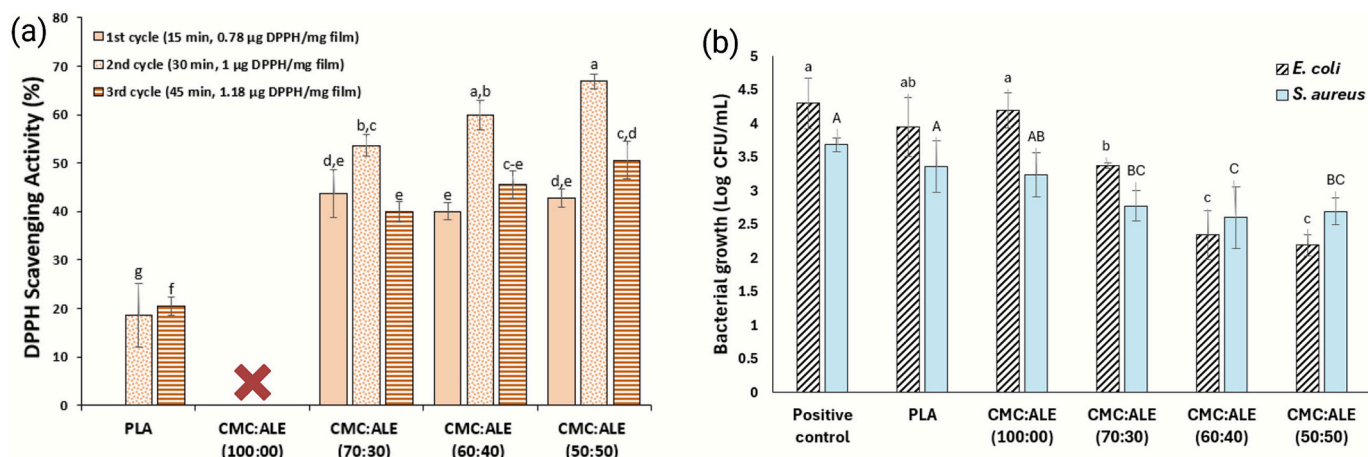


Fig. 5. (a) Antioxidant activity (DPPH scavenging) of plain PLA and coated with various ratios of carboxymethyl cellulose (CMC) and artichoke leaf extract (ALE) over three cycles of DPPH addition; (b) antibacterial activity of the same coatings against *E. coli* and *S. aureus* (Different letters indicate significant differences among mean values of each parameter across different groups ( $P < 0.05$ )).

differences among different formulations. The antibacterial activity of ALE might be attributed to phenolic compounds, which interact with bacterial cell membranes and by changing the membrane permeability causing leakage of cellular contents such as proteins, nucleic acids, ions, etc. Other antimicrobial mechanism can be related to their potential to induce oxidative stress so that phenolics can generate ROS or disrupt redox homeostasis, causing oxidative damage to membranes, proteins, and DNA [34]. The observed inhibition of both Gram-positive and Gram-negative bacteria highlights ALE as an effective antimicrobial agent, consistent with previous studies [30,47].

### 3.5. Kinetics of the polyphenols release

The release of polyphenols from coating materials into three food simulants over time is shown in Fig. 6. Generally, the release of phenolics is governed by several factors, including the penetration of the simulants into the coating matrix, swelling of the coating matrix, solubilization of phenolic compounds, and their diffusion through the polymer matrix into the surrounding medium [9,11,53].

Polyphenols concentrations increased over time in all three simulants, but the release patterns differed among them. In 50 % ethanol, an initial burst release was observed, followed by a slower, sustained phase. In contrast, 10 % ethanol exhibited a slower initial release, followed by a rapid burst, while 3 % acetic acid showed a relatively sustained release throughout the testing period. Compared with hydrophilic simulants (10 % ethanol and 3 % acetic acid), the lipophilic simulant (50 % ethanol) facilitated faster polyphenol release. This behavior may be attributed to the method used for extraction of ALE (subcritical water

extraction), which extracts both polar and nonpolar compounds, and the higher solubility of these compounds in 50 % ethanol.

During the early release phase in hydrophilic simulants, the release was concentration-dependent. This can be explained by the hydrophilic nature of CMC and its higher solubility in 10 % ethanol and 3 % acetic acid, which promotes exposure and rapid diffusion of polyphenols. Subsequently, the release rate slowed in formulations with higher ALE content, likely because more time was required for the complete release of phenolics. In 50 % ethanol, higher ALE concentrations showed slower release due to the limited solubility of CMC in this medium, requiring additional time for matrix swelling and release.

Release kinetics were fitted using different models, with coefficients summarized in Tables S1–4. In 50 % ethanol, the Korsmeyer–Peppas (Power Law) model provided the best fit ( $R^2 > 0.95$ ), while in 10 % ethanol and 3 % acetic acid, the Weibull model showed a better fit ( $R^2 > 0.93$ ). For the Weibull model, b values greater than 1 indicate a complex release mechanism. According to the Korsmeyer–Peppas model, an n value  $< 0.45$  corresponds to Fickian diffusion,  $n > 0.89$  indicates a swelling-controlled mechanism, and n values between 0.45 and 0.89 suggest a combination of diffusion and swelling [25,28]. Therefore, in the lipophilic simulant (50 % ethanol), polyphenol release was controlled by both diffusion and swelling, whereas in hydrophilic media (10 % ethanol and 3 % acetic acid), the release was predominantly swelling-controlled.

### 3.6. Fruits preservation performance

Two formulations of coating including 50:50 and 60:40 (CMC:ALE)

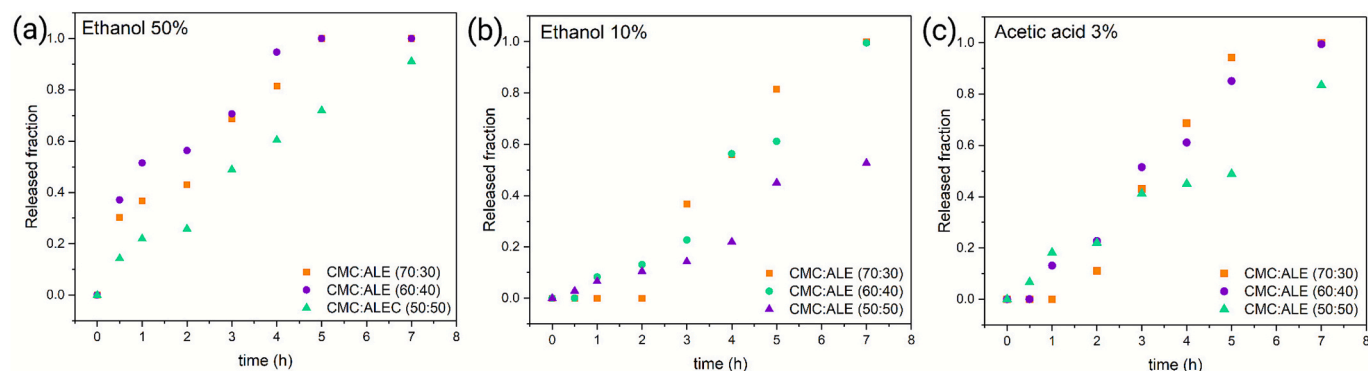


Fig. 6. Percentage of polyphenols released to different food simulants: (a) ethanol 50 %, (b) ethanol 10 %, and (c) acetic acid 3 %.



showed similar performance in terms of bioactivity, however due to better stability of 60:40 (CMC:ALE) coating and lower moisture absorption, this packaging was selected for further preservation studies on fresh-cut apples and strawberries. The performance of the coated packaging was compared with that of the same packaging without coating and a control group without any packaging.

### 3.6.1. Cut apples quality

Fresh-cut apples are popular for their convenience, nutrients (vitamins, fiber, minerals), and disease resistance, but they deteriorate quickly during storage due to oxidation and nutrient loss. To evaluate coated packaging, physicochemical properties and microbial loads were analyzed. Visual monitoring over 8 days (Fig. 7a) showed all groups looked similar by day 2. By day 4, unpackaged apples showed pronounced oxidation and deterioration, while uncoated and coated groups displayed milder changes. After 8 days, unpackaged apples exhibited severe oxidation and decay, whereas uncoated and coated groups showed less deterioration.

Weight loss significantly affects fruit quality, with rapid moisture loss accelerating decay and better retention maintaining freshness. By day 8, unpackaged apples had the highest weight loss (3.86 %), while uncoated and coated packaging showed minimal loss (<0.6 %) with no significant difference (Fig. 7b). Titratable acidity, reflecting sugar-acid balance and overall metabolism, decreased in all groups, with irregular trends in unpackaged and uncoated groups (Fig. 7c). The greatest reductions occurred in unpackaged (20.74 % on day 8) and uncoated groups (17.82 % on day 6), whereas coated packaging showed only an 11.64 % reduction, indicating higher stability by limiting oxygen exposure and respiration. pH gradually increased in all groups with some fluctuations (Fig. S4), likely due to gram-negative bacterial growth and anaerobic metabolite production, as previously reported [40]. The highest pH was observed in unpackaged apples, reaching 3.53 on day 6, while the coated group showed minimal change from 3.35 to 3.47, indicating better quality preservation.

Total phenolic content (TPC) increased until day 6 (Fig. 7d), likely

due to cell wall disruption, enhanced phenolic synthesis, enzymatic activity, and migration of phenolics from the coating [26], then declined due to oxidation and polymerization. Phenylalanine ammonia-lyase (PAL) and PPO enzymes contributed to these TPC changes by biosynthesis and oxidation of phenolic compounds, respectively.

In addition, the antioxidant potential of cut apples during storage assessed by % DPPH scavenging decreased by 40.82, 25.83, and 21.98 % in the unpackaged, uncoated, and coated groups, respectively compared to initial activity of 63.27 % (Fig. S5). Packaging, particularly coated packaging, helped slow down the oxidation and polymerization of phenolic compounds, thereby better preserving antioxidant activity. Similar effects were reported with chitosan-based films containing Nickel Ferrite nanoparticles, which preserved DPPH scavenging at 36 % compared to 10 % in controls [15].

Fruit firmness, a key quality attribute, was also affected by storage conditions (Fig. 7e). Unpackaged apples lost the most firmness (49.24 % by day 8) due to cellulose and pectin degradation [42], whereas uncoated and coated groups showed smaller reductions of 36.52 % and 37.13 %, respectively, with no significant difference between them.

Color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) and browning index (BI) were also evaluated (Table 1).  $L^*$  value, representing brightness, decreased in all groups, but less in packaged apples, while  $a^*$  and  $b^*$ , representing the green-red and blue-yellow color components respectively, increased, reflecting surface darkening of sample surface toward a redder and yellower appearance, although it was less pronounced in packaged apples. BI values quantifying surface browning, after 2 days were 57.07 (unpackaged), 61.73 (uncoated), and 49.82 (coated), indicating the coating slowed oxidation. By day 8, packaging reduced oxygen exposure and enzymatic browning, though differences between uncoated and coated groups were minimal, suggesting limited coating effect after 48 h. Coated packaging performed better than chitosan quaternary ammonium films with copper-ammonia fiber/cinnamaldehyde (which delayed browning by ~14 h) [43], however direct coating of apple, such as ferulic acid-gelatin edible films, could provide superior browning protection [42].

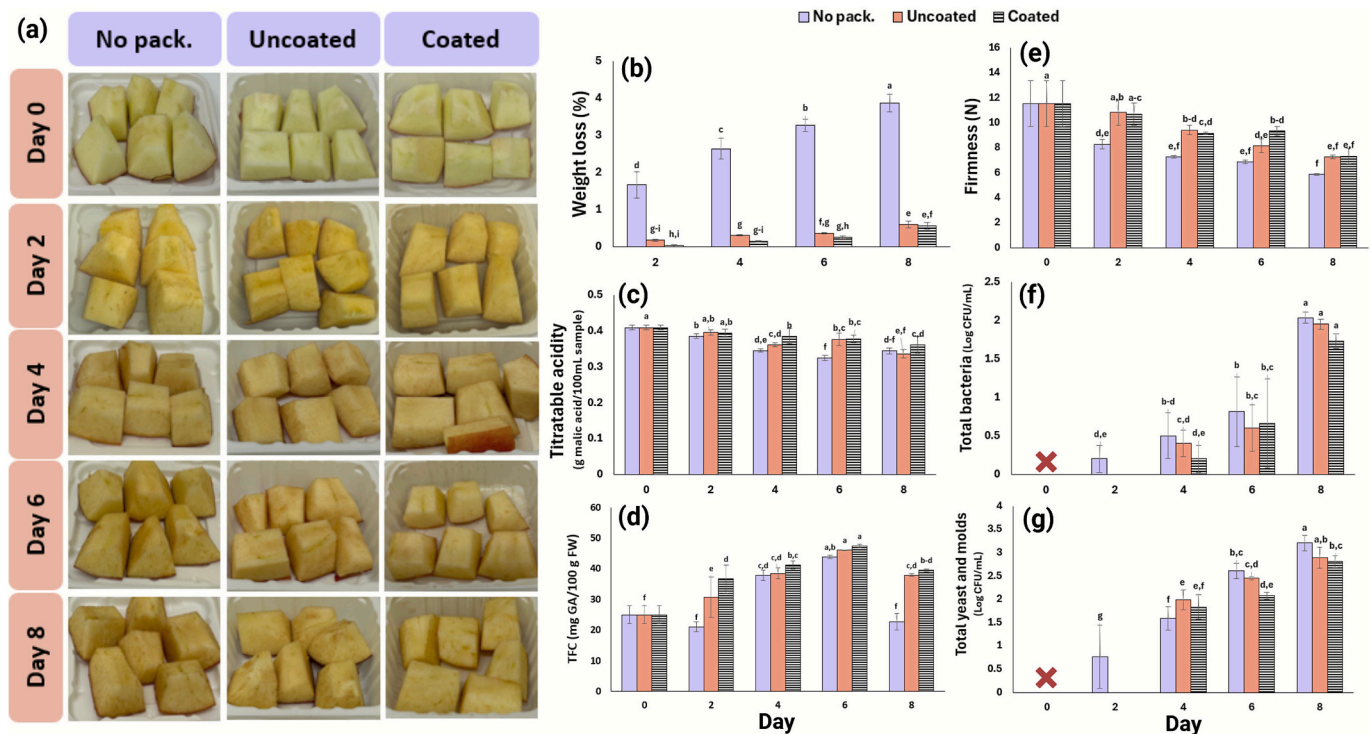


Fig. 7. (a) Visual changes and variation in (b) weight loss, (c) titratable acidity, (d) total phenolic content, (e) firmness, (f) total viable bacteria, and (g) total mold and yeast of cut apples stored at 4 °C for 8 days (Different letters indicate significant differences among mean values of each parameter across different groups ( $P < 0.05$ )).



**Table 1**

Color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) and the browning index (BI) of cut apples stored at 4 °C for 8 days.

Time (Day)	$L^*$	$a^*$	$b^*$	BI
No packaging				
0	75.49 ± 2.10 <sup>a</sup>	−3.96 ± 0.76 <sup>f</sup>	31.46 ± 1.93 <sup>f</sup>	46.27 ± 2.44 <sup>f</sup>
2	73.97 ± 4.05 <sup>a-c</sup>	0.56 ± 0.69 <sup>d</sup>	34.24 ± 1.62 <sup>c-e</sup>	57.07 ± 4.68 <sup>c</sup>
4	74.78 ± 4.05 <sup>a,b</sup>	1.25 ± 0.18 <sup>c</sup>	32.38 ± 1.27 <sup>d-f</sup>	55.55 ± 3.25 <sup>d,e</sup>
6	67.51 ± 2.58 <sup>d</sup>	0.93 ± 0.16 <sup>a</sup>	37.91 ± 2.35 <sup>a,b</sup>	53.57 ± 3.30 <sup>c-e</sup>
8	66.92 ± 1.79 <sup>d</sup>	4.41 ± 0.94 <sup>a</sup>	39.08 ± 2.74 <sup>a</sup>	81.89 ± 1.13 <sup>a</sup>
Uncoated packaging				
0	75.49 ± 2.10 <sup>a</sup>	−3.16 ± 0.76 <sup>f</sup>	31.46 ± 1.93 <sup>f</sup>	46.27 ± 2.44 <sup>f</sup>
2	72.53 ± 2.42 <sup>b,c</sup>	0.96 ± 0.57 <sup>c</sup>	35.41 ± 2.45 <sup>b,c</sup>	61.73 ± 6.34 <sup>c</sup>
4	71.60 ± 1.92 <sup>c</sup>	0.96 ± 0.94 <sup>c</sup>	34.80 ± 1.56 <sup>c</sup>	62.76 ± 6.13 <sup>c</sup>
6	73.08 ± 2.96 <sup>a-c</sup>	1.61 ± 0.61 <sup>c</sup>	33.63 ± 2.79 <sup>c-f</sup>	56.96 ± 5.32 <sup>c</sup>
8	71.69 ± 2.15 <sup>c</sup>	2.87 ± 0.33 <sup>b</sup>	38.38 ± 2.80 <sup>a</sup>	71.51 ± 4.17 <sup>b</sup>
Coated packaging				
0	75.49 ± 2.10 <sup>a</sup>	−3.16 ± 0.76 <sup>f</sup>	31.46 ± 1.93 <sup>f</sup>	46.27 ± 2.44 <sup>f</sup>
2	75.55 ± 0.57 <sup>a</sup>	0.03 ± 0.50 <sup>e</sup>	31.58 ± 1.91 <sup>e,f</sup>	49.83 ± 1.70 <sup>e,f</sup>
4	72.92 ± 0.89 <sup>a-c</sup>	0.87 ± 0.14 <sup>c</sup>	34.17 ± 1.07 <sup>c-f</sup>	61.08 ± 2.87 <sup>c-e</sup>
6	73.01 ± 2.33 <sup>a-c</sup>	0.62 ± 0.20 <sup>d</sup>	33.19 ± 2.33 <sup>c-f</sup>	55.49 ± 3.47 <sup>c</sup>
8	72.06 ± 0.93 <sup>b,c</sup>	2.60 ± 0.34 <sup>b</sup>	38.13 ± 3.00 <sup>a,b</sup>	71.26 ± 3.66 <sup>b</sup>

$L^*$  indicates lightness (0 = black, 100 = white),  $a^*$  represents the red-green axis (positive = red, negative = green), and  $b^*$  represents the yellow-blue axis (positive = yellow, negative = blue).

Different letters (a–f) indicate significant differences among mean values of each parameter across different groups ( $P < 0.05$ ).

Microbial spoilage is the primary factor limiting the shelf life of fresh-cut fruits and vegetables. Microbial growth during the storage period is shown in Fig. 7f and g. Initially, the freshly cut apples were almost free of microbial contamination. In the packaged groups, both coated and uncoated, microbial loads remained negligible up to day 2. In contrast, the without packaging group already exhibited microbial growth, with counts of 0.2 log CFU for total bacteria and 0.77 log CFU for total yeast and mold. From day 4 onward, the populations of bacteria, yeasts, and molds increased in all groups, although the increase was less pronounced in the coated group on certain days. By the end of storage, the coating significantly reduced total yeast and mold counts compared to the without packaging group; however, it showed an insignificant decrease in bacterial growth. This outcome may be attributed to the reduced oxygen and moisture exchange in packaging. While this slows some aerobic bacteria, it may create microenvironments conducive to the growth of facultative anaerobes. Condensation inside the package can further enhance microbial proliferation by increasing local water activity.

### 3.6.2. Strawberries quality

Strawberries are highly susceptible to deterioration due to physiological respiration, microbial contamination, and physical damage. In addition, their active transpiration and respiration after harvest accelerate deterioration, resulting in a short shelf life [42]. Fig. 8a illustrates the changes in appearance of strawberries stored at 4 °C over 10 days. By day 10, strawberries without packaging showed evident shrinkage and color change caused by mold growth. In comparison, fruits stored in uncoated packaging exhibited texture breakdown and juice leakage, with black spots induced by mold growth. Conversely, strawberries

stored in coated packaging maintained better appearance, with no visible signs of spoilage.

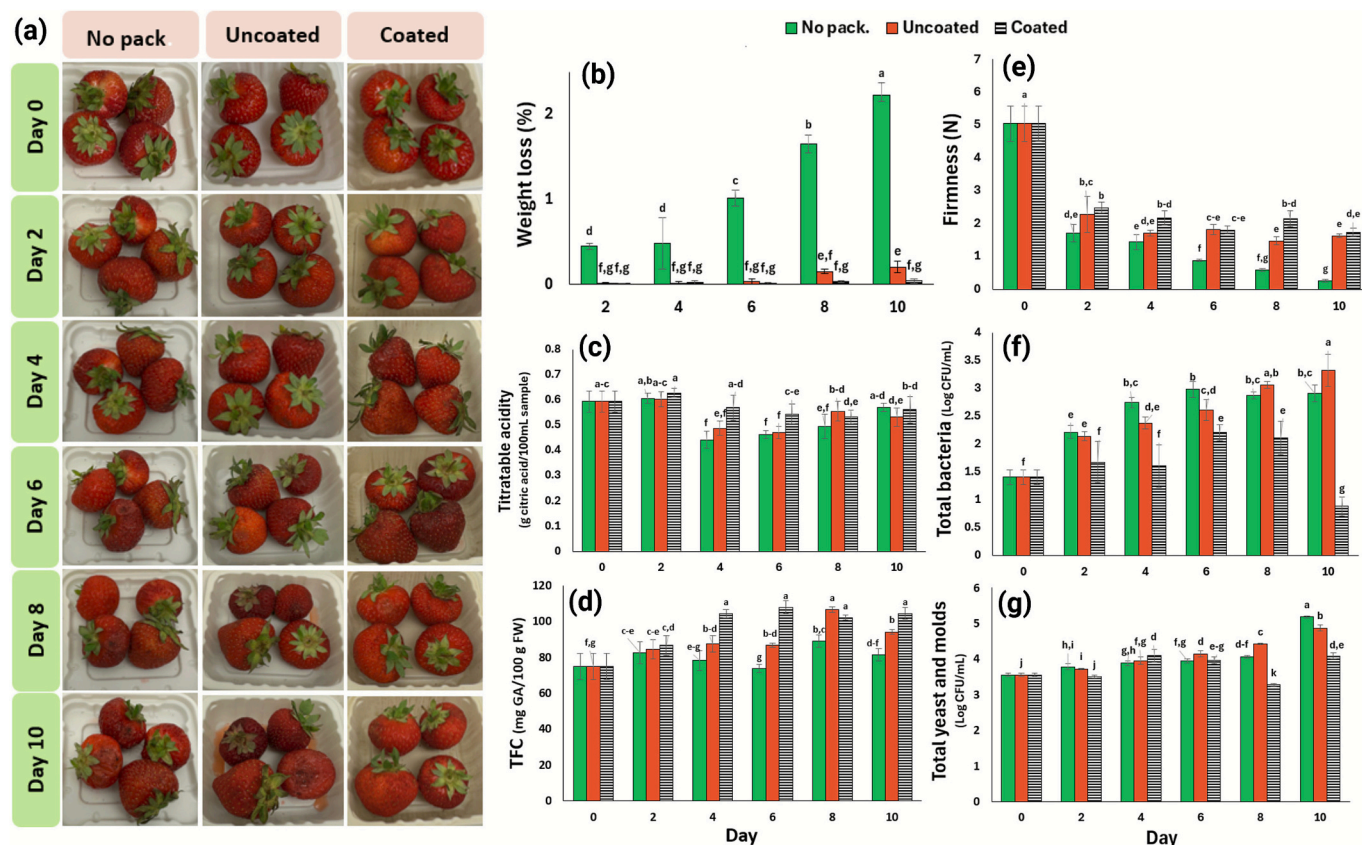
Strawberry weight loss gradually increased during storage (Fig. 8b), with unpackaged fruits losing the most (2.21 % by day 10), while uncoated and coated groups showed minimal loss (<0.3 %), with a significant difference on day 10 due to the lower WVP of coated packaging.

Titrateable acidity of all three groups of strawberries generally exhibited a decline in acidity during storage, although the trend was irregular (Fig. 8c). This reduction is commonly observed in fruit preservation and is mainly attributed to the consumption of organic acids as respiratory substrates [5,14]. The control group showed the largest change (0.44–0.59 %), reflecting a high respiration rate and metabolic activity in the absence of preservation measures. In the uncoated group, the change was smaller (0.47–0.59 %), indicating that uncoated packaging partially suppressed respiration and organic acid degradation. By contrast, the coated group maintained a relatively stable acidity (0.54–0.62 %), which may be explained by improved regulation of respiration, slower metabolic consumption of organic acids, and restricted microbial growth. These results align with the superior water vapor barrier and antimicrobial properties of the coated packaging and are consistent with previous findings on strawberry preservation using *Schizochytrium limacinum* oil-based Pickering emulsions in a chitosan film [56]. pH generally decreased with fluctuations in all strawberry groups (Fig. S6) due to progressive acidification from microbial activity and metabolic changes [16]. Compared to unpackaged fruits, uncoated (3.52–3.45) and coated (3.52–3.43) groups showed smaller pH variations, indicating that packaging effectively stabilized the internal acid-base environment by slowing respiration and metabolic activity.

Strawberry TPC initially increased then declined (Fig. 8d), with the lowest TPC in unpackaged strawberries on day 6, similar to trends in low-density polyethylene (LDPE) stored fruit [35]. Phenolic compounds, including phenolic acids, flavonoids, and anthocyanins, play key roles in defense against stress, ROS, and microbes while maintaining fruit quality [27], but are prone to PPO-mediated oxidation. Packaging with strong oxygen-barrier properties limits phenolic degradation, and maintaining cell wall integrity, firmness, and antioxidant capacity further stabilizes phenolics [36]. Coated packaging effectively minimized phenolic loss, likely due to oxygen-barrier performance and better quality preservation. Strawberries retained over 91 % of antioxidant activity across all groups (Fig. S7), with DPPH scavenging slightly declining due to oxidative degradation. Antioxidant activity remained highest in the coated group throughout storage, highlighting both the coating's protective effect and possible migration of ALE phenolics to the fruit surface.

Strawberry firmness decreased during storage due to cell wall disruption and polysaccharide depolymerization from respiration and microbial activity [52]. Firmness dropped sharply by day 2 and then declined gradually in all groups (Fig. 8e). The coated group retained the most firmness, likely due to ALE's antibacterial effect, which limited microbial proliferation and helped preserve cell wall structure.

Strawberries are highly prone to microbial spoilage. Bacterial, mold, and yeast populations increased during storage in unpackaged and uncoated groups (Fig. 8f and g). In contrast, the coated group demonstrated clear antimicrobial efficacy, particularly against bacteria, where growth was suppressed throughout storage, resulting in a significant 37.29 % reduction compared to day 0. The coated group also inhibited molds and yeasts from day 8 onward, achieving a 1.09 log reduction compared to unpackaged fruit by day 10. Interestingly, the uncoated group exhibited higher microbial contamination than unpackaged fruits. This can be explained by limited gas exchange creating favorable conditions for both aerobic and anaerobic microbial growth as well as the moisture condensation on the inner surfaces and created localized high-humidity zones favorable for microbial proliferation. Additionally, surface contact among fruits and the packaging may have facilitated cross-contamination.



**Fig. 8.** (a) Visual changes and variation in (b) weight loss, (c) titratable acidity, (d) total phenolic content, (e) firmness, (f) total viable bacteria and (g) total mold and yeast of strawberries during storage at 4 °C for 10 days (Different letters indicate significant differences among mean values of each parameter across different groups ( $P < 0.05$ )).

#### 4. Conclusions

This study highlights the potential of food loss streams as valuable resources for developing active coatings for PLA-based packaging within a sustainable circular bioeconomy framework. ALE, derived from food loss, was combined with CMC to obtain a coating solution with improved viscosity and flowability. Uniform coating deposition was achieved only after surface pre-treatment of PLA with a PLA-PEG layer, which enhanced surface hydrophilicity and interfacial adhesion with the active coating. The resulting thin active coating ( $\sim 5 \mu\text{m}$ ) imparted notable antioxidant and antibacterial properties to the PLA-based packaging. Among the tested formulations, the CMC:ALE (60:40) ratio provided the best balance between bioactivity, moisture resistance, and coating uniformity. Release kinetics modeling indicated that polyphenol release in lipophilic media (50 % ethanol) was governed by both diffusion and swelling, whereas in hydrophilic media (10 % ethanol and 3 % acetic acid), it was primarily swelling-controlled. Shelf-life studies demonstrated that this active packaging effectively preserved the freshness of highly perishable fruits such as strawberries by reducing microbial growth and maintaining phenolic content and antioxidant activity. In contrast, its effect was less pronounced for cut apples, likely due to their different physiological and preservation characteristics. Despite these promising results, the hydrophilic nature of the coating presents a limitation under high-moisture conditions, as condensation inside non-ventilated packaging may lead to localized swelling or partial solubilization of the coating after extended storage. Therefore, future work should focus on enhancing the water resistance and long-term stability of the coating without compromising its bioactive functionality.

#### CRediT authorship contribution statement

**Zeinab Qazanfarzadeh:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Amparo Jiménez-Quero:** Writing – review & editing, Writing – original draft, Validation, Supervision, Project administration, Funding acquisition, Conceptualization.

#### Declaration of competing interest

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

Amparo Jimenez Quero reports financial support was provided by Chalmers University of Technology. 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.

#### Data availability

Data will be made available on request.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.susmat.2025.e01783>.

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