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The Use of Bioactive Edible Coatings Based on Pectin and Phenolic Acids for Enhancing Quality Attributes of Golden Delicious Apples During Storage

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Abstract

This research study investigated the effect of edible coatings made from apple pectin, incorporating caffeic and protocatechuic acids, on the quality attributes of Golden Delicious apples during 28 days of storage at ambient conditions. The study evaluated the rheological properties of the coating solutions, the release of phenolic acids from the edible films, and various quality characteristics of the apples. These characteristics included weight loss, colour, total soluble solids, total titratable acidity, pH, firmness, respiration rate, ripeness level, and sensory analysis. The results showed that all coating solutions exhibited non-Newtonian, shear-thinning flow behaviour, with the sample containing protocatechuic acid demonstrating a decrease in apparent viscosity. Additionally, both phenolic acids were released rapidly from the film into a 96% ethanol medium. The study found that bioactive edible coatings, both with and without phenolic acids, were significantly effective in reducing weight loss, colour changes, firmness, and ripening of apples during storage. The total soluble solids were higher in control apples (14.95 ± 0.48 °Brix at 28 days) compared to the coated samples (13.52–13.53 °Brix at 28 days), indicating that the control apples were riper and contained a higher amount of sugars. Ethylene production decreased after 4 weeks of storage, from 60.40 ppm for the apples before storage to 23.55–25.70 ppm for the coated samples, and only to 52.75 ppm for the control apples. Overall, this study confirmed that the use of developed bioactive coatings extends the shelf life of apples by preserving their quality and sensory attributes during storage.

Keywords: edible coatings; Golden Delicious apple; apple pectin; phenolic acids; colour changes; firmness; ripening; sensory analysis

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1. Introduction

Effectively managing post-harvest fruit poses numerous challenges that must be addressed to preserve fruit quality during extended storage under various conditions [1]. Apples are classified as climacteric fruits, meaning they continue to ripen after being harvested. This ongoing metabolic activity makes them sensitive to quality loss. To preserve their desired quality, apples are typically stored in cold storage or in a controlled atmosphere for long-term storage [2,3]. Apples are typically harvested in August and September, but they are available for sale and consumption throughout the entire year. To ensure that consumers have access to apples during the off-season, they are often stored for 4 to 6 months [4]. This is particularly advantageous in regions where fruit is only available

or cultivated during specific seasons. To address the challenge of storing and preserving apples after harvest, it is essential to develop a new preservation method, particularly for fruits stored at room temperature. Cold storage can negatively affect the texture, flavour, and colour of fruit. Thus, edible coating offers a better solution by protecting against gases, moisture, pathogens, and light [5].

In recent years, there has been a growing interest in environmentally friendly edible coatings that can extend the shelf life of fresh fruits and vegetables [6–8]. Edible coatings are thin layers of material applied to products, typically through immersion [9]. These coatings act as a semi-permeable membrane, providing protection against moisture, oxygen, and carbon dioxide. They can help reduce the rate of oxidation reactions, respiration, and moisture loss in the products, thereby prolonging their storage period [10,11]. Apples, due to their biological activity after harvest, become increasingly susceptible to infections caused by pathogenic fungi, such as *Penicillium expansum*, *Penicillium digitatum*, or *Botrytis cinerea*, as they ripen and age. These fungi are responsible for post-harvest decay [12–14]. Commercial storage can negatively impact the emission of apple aroma, which may persist throughout the retail chain, resulting in poor consumer acceptability [15]. The use of coatings reduces undesirable changes in fruit, preserving a fresh appearance that is characterised by good quality and sensory appeal [16]. Research indicates that nearly 71% of fruit is consumed fresh, while around 20% is processed into various value-added products [10]. Effective storage is essential to reduce post-harvest losses of fresh apples, which can reach 25–28%. New solutions need to be developed to extend the shelf life of fruits and increase the food supply [17]. Edible coatings have emerged as a highly effective and safe technology for preserving fresh fruits. They work by limiting gas exchange, which delays the ageing process of fruits during storage [6,18,19]. Edible coatings containing antimicrobial compounds, such as plant extracts or essential oils, inhibit the growth of microorganisms that can lead to the spoilage of fruits [20]. Many coatings are enriched with antimicrobial compounds or chitosan, which directly inhibit bacterial and fungal growth by disrupting cell membranes or carrying out other direct antimicrobial actions [21].

Edible coatings can be made from proteins, polysaccharides, lipids, or a combination of these ingredients. Additionally, edible coatings can serve as carriers for various food additives, including antimicrobial agents, anti-browning agents, antioxidants, as well as dyes, flavours, nutrients, and spices [22,23]. The main purpose of applying edible coatings to fresh fruit is to reinforce the existing natural barrier or replace it where it has been partially removed. Other benefits of edible coatings include improved mechanical properties by maintaining the structural integrity of coated products. By allowing the coatings to be consumed with fruit, it is possible to reduce packaging waste [24]. However, the disadvantages include the formation of an unpleasant aftertaste if the coating is too thick, as well as the occurrence in some cases of a hygroscopic nature, which favours the growth of microorganisms [25]. Different types of polysaccharides, like pectin, are commonly used to develop edible coatings for the preservation of various fruits. Pectin coatings offer excellent resistance to moisture and gases and are highly transparent, helping to preserve the sensory qualities and overall quality of the fruit [5,26]. Edible coatings that contain lipids can substantially reduce moisture loss in fruits and contribute to a lighter skin colour. These coatings serve as semipermeable membranes, which limit the flow of gases and water vapour [27]. As a result, they slow down the rates of respiration and moisture loss in fruits, helping to maintain fruit quality and delay physiological deterioration after harvest [28]. The estimated growth in biopolymer production, the main components of edible coatings, is expected to reach up to 2.41 million tons by 2030 [5]. The rising popularity of using natural polymers for coating and preserving food can be attributed to their potential benefits and the fact that they can be safely consumed alongside

fruits. Furthermore, unlike synthetic or petroleum-based coatings, natural polymers are environmentally friendly and help reduce packaging waste, aligning with the principles of sustainable development [21,29].

Malus domestica Borkh., known as Golden Delicious apples, is among the most commonly consumed and cultivated apple varieties in temperate regions of the world. They are valued for their shape, texture, nutritional benefits, and excellent taste [30,31]. They are prone to post-harvest softening because they ripen in summer [32]; however, for optimal storage, the conditions of 0–4 °C and 90–95% relative humidity, with a maximum storage duration of 2 to 6 months, are indicated [33]. Their storage life is estimated to be approximately four weeks [17]. Proper storage of apples is essential for economic processes, as it minimises food waste and promotes more sustainable food consumption [34].

Pectin is an effective coating material for protecting the quality of fresh apples. It can also serve as a carrier for functional ingredients, thereby enhancing antioxidant and antimicrobial properties [35]. Pectin's amphiphilic properties can improve stability by reducing surface tension. The presence of methoxy groups influences the hydrophobic nature of pectin, endowing it with surface-active properties. Furthermore, coating fresh fruit with edible coatings helps maintain firmness due to the coatings' antagonistic effect on microflora, which can contribute to the progressive softening of tissue [36]. Studies have shown that edible coatings and films are more effective in extending the shelf life of fresh produce when formulated as composite formulations with other ingredients, rather than using a single component [28]. Various coatings were applied to fresh apples to extend their shelf life. De Léon-Zapata et al. [37] observed that applying a candelilla wax-based edible coating, which included fermented tarbush extract as a natural antioxidant source, positively affected the quality and shelf life of Golden Delicious apples. Rashid et al. [38] demonstrated that composite coatings containing 2.5 g of fenugreek and 1.5 g of flaxseed polysaccharides, combined with stearic acid, monoglycerides, and canola oil, were the most effective for maintaining fruit mass, firmness, total soluble solids, acidity, and pH of apples. The effectiveness of these coatings can be attributed to their ability to delay the respiration rate and inhibit ethylene production in the fruit. Soppelsa et al. [9] observed that thyme and clove essential oils, when encapsulated in chitosan coatings, effectively control postharvest diseases of apple fruits stored at 20 °C and 100% relative humidity. Conversely, the quality of fresh-cut apples, which are prone to rapid deterioration, may also be extended by bioactive coatings. In this context, Nicolau-Lapeña et al. [39] noted the beneficial effect of the incorporation of ferulic acid into sodium alginate-based coatings in a reduction in browning and the population of *Listeria monocytogenes* after 7 days of refrigerated storage of apple slices.

The findings indicate that encapsulated active ingredients can be a valuable tool for managing apples after harvest. However, before widespread application, we must further investigate the economic sustainability of available biopolymers and their potential negative impact on fruit aroma and taste. Phenolic acids act as antioxidants by neutralising free radicals, thereby protecting against oxidative damage. Their antioxidant properties stem from a hydroxyl group attached to an aromatic ring, which allows them to donate a hydrogen atom to stabilise free radicals [40]. Abundant in fruits, vegetables, and beverages, they are considered a major class of natural antioxidants in the human diet and have potential applications in medicine, cosmetics, and the food industry [41]. Caffeic acid is a promising antioxidant that can be incorporated into edible coatings to protect food by scavenging free radicals, chelating metal ions, and preventing lipid oxidation. Research indicates that the addition of caffeic acid enhances antioxidant activity, UV protection, and antimicrobial properties in films composed of biopolymer-based materials [42]. Protocatechuic acid is another effective antioxidant compound for edible packaging, enhancing the film's ability

to protect food from oxidative damage and serving as a UV light barrier [43]. The addition of phenolic acids to film-forming solutions can improve properties like flexibility, while also helping to extend the shelf life of different food products by delaying spoilage.

The objective of this study was to preserve the quality of fresh Golden Delicious apples by applying a pectin coating that included selected phenolic acids, specifically caffeic and protocatechuic acids, during storage at room temperature. To assess the effectiveness of the edible coating treatment, several parameters were measured over a 28-day period while storing the apples at room temperature. These parameters included changes in weight, colour, total soluble solids, titratable acidity, pH, firmness, respiration rate, degree of ripeness, and sensory attributes. Further research is required to investigate the combination of caffeic acid and protocatechuic acid formulations and determine any additive or synergistic effects on antioxidant or antimicrobial activity, including release kinetics. It may also be important to determine the likelihood of antagonistic interactions resulting from the combination of these two phenolic acids.

2. Materials and Methods

2.1. Materials

Apple pectin (Pektowin S.A., Jasło, Poland) and caffeic (Pol-Aura Sp. z o.o., Zawroty, Poland) or protocatechuic acids (Thermo Scientific, Gdańsk, Poland) were used for the production of aqueous coating solutions. Glycerol (Avantor Performance Materials Poland S.A., Gliwice, Poland) was used as a plasticising agent. Golden Delicious apples from the experimental orchards of the Warsaw University of Life Sciences were harvested in 2024 and used for experiments.

2.2. Preparation of Coating Solutions

The selected phenolic acids were combined with apple pectin before hydration at a concentration of 5% (acids relative to pectin). The solutions were heated at 60 °C for 20 min using an RCT basic IKAMAG magnetic stirrer (IKA Poland, Warsaw, Poland) with a rotation speed of 600 rpm to obtain a uniform solution. After cooling the solutions, glycerol was added at a concentration of 50% relative to apple pectin (2.5 g). For the experiments, three coating solutions based on apple pectin were used: one without phenolic acids, coded AP; one with caffeic acid, coded AP_CFA; and one with protocatechuic acid, coded AP_PCA. Distilled water was used instead of the coating solution for control samples. The characteristics of the substances used and their proportions are presented in Table 1.

Table 1. Characteristics of the substances used to obtain film-forming solutions.

Substance	Quantity (g)
Apple pectin (AP)	5
Caffeic acid (CFA)	0.25
Protocatechuic acid (PCA)	0.25
Glycerol (GLY)	2.5
Water	92.25

Rheology of Coating Solutions

A Haake MARS 40 rheometer (Thermo Scientific Inc., Waltham, MA, USA) was used to study the flow behaviour of the solutions at 25 °C in triplicate in a coaxial cylinder

system (CC25DIN/Ti) with a linearly increasing shear rate up to 100 s^{-1}. The flow curves were fitted using the Ostwald de Waele model [44]:

$$\tau = K \cdot \dot{\gamma}^{n}$$

where τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), K is the consistency index (Pa·s^n), and n is the dimensionless flow behaviour index.

2.3. Film Preparation

The films were obtained based on the method described in our previous study [45]. Briefly, the coating solutions were poured onto sheets at a speed of 10 mm/s and a layer thickness of 2500 µm using a Zehntner ZAA 2300 automatic film applicator (Zehntner GmbH Testing Instruments, Sissach, Switzerland) and dried in a laboratory dryer SUP-65W (Wamed, Warsaw, Poland) for 24 h at 50 °C. The obtained films were conditioned in a KFB 240 thermostatic chamber (Binder, Tuttlingen, Germany) at 25 °C and 50% relative humidity for 48 h prior to testing.

2.3.1. Film Thickness

The film thickness was determined at least in three replicates, and the values were used to determine the kinetics of phenolic acid release. A thickness tester, ProGage (Thwing-Albert, West Berlin, NJ, USA), with an accuracy of 1 µm, was used.

2.3.2. The Release Kinetic Measurements of Phenolic Acids

Phenolic acid release was conducted in three replicates using a 96% ethanol solution (Chempur, Piekary Śląskie, Poland) based on the method described by Benbettaieb et al. [46]. Developed films dissolved instantly in water, so 96% ethanol was used to evaluate release over time. Film samples weighing approximately 60 ± 5 mg were placed in beakers, which were then filled to a total volume of 100 mL with ethanol. The prepared samples were stirred at 150 rpm using an RCT basic IKAMAG magnetic stirrer (IKA Poland, Warsaw, Poland). Absorbance measurements were taken using an Evolution 220 UV–Visible Spectrophotometer (Thermo SCIENTIFIC, Warsaw, Poland). For one hour, 4 mL of the solution containing the released substances was collected at specific time intervals: every minute for the first 10 min, and every 5 min for the remaining duration. The concentrations of phenolic acids in the samples were determined by UV-VIS spectrophotometry at wavelengths of 310 nm for caffeic acid and 315 nm for protocatechuic acid. After each measurement, the collected solution was returned to the beaker to maintain a constant volume. The calibration curve was prepared for each compound at concentrations from 0.5 to 2.5 mg/100 mL of 96% ethanol, giving the equations of y = 0.9637x + 0.2184 (R^2 = 0.944) for caffeic acid and y = 0.805x − 0.0511 (R^2 = 0.973) for protocatechuic acid. The obtained results were used to calculate the release of active substances using Fick's second law equation in the transient state [46,47]:

$$\frac{Ct}{C\infty} = 1 - \sum_{n=1}^{\infty} \frac{2\alpha(1+\alpha)}{1+\alpha+\alpha^2 q_n^2} \exp\left(-\frac{D q_n^2 t}{L^2}\right)$$

where C_t (mg/L) is the concentration of the active substance over time in the release medium; C_∞ (mg/L) is the concentration of the active substance at equilibrium in the release medium; D is the diffusion coefficient of the active substance (m^2/s); L is half the thickness of the film (m); q_n are non-zero, positive roots of $\tan(qn) = -\alpha qn$ (n value from 1 to 6); α is determined on the basis of $\alpha = \frac{V_s}{K_{f,S} x V_f}$; V_f, film volume (m^3); $K_{f,S} = \frac{C_{f,\infty}}{C_{S,\infty}}$: volume of the medium, and the partition coefficient of the active substance between the foil and the simulant (solution) in equilibrium; $C_{f,\infty}$ and $C_{S,\infty}$ are the equilibrium concentrations of the active substance (mg/L) in the film and the food simulant, respectively.

2.4. Coatings of Apples

Apples of similar size and ripeness were washed with tap water, followed by drying using a paper towel. The apples were then immersed in coating solutions for 15 s, followed by a 5 s immersion in a 1% calcium chloride solution (Avantor Performance Materials Poland S.A., Gliwice, Poland) for pectin cross-linking. Control apples were immersed in distilled water for 20 s. Coated fruits were blotted to remove excess solution, placed on filter paper, and stored at room temperature (22 ± 1 °C) and 40 ± 5% relative humidity for 28 days. Testing occurred at 7-day intervals. There were four variants of samples in three repetitions: control apples treated with water (Control), apples coated with solutions without phenolic acids (AP), and apples coated with solutions containing caffeic (AP_CFA) and protocatechuic (AP_PCA) acids. Each sample of the variant consisted of three apples per repetition, with an average weight of 150 ± 20 g, for which separate analyses were performed at 0, 7, 14, 21, and 28 days post-treatment.

3. Physicochemical Analyses

3.1. Weight Loss

The determination of weight loss in all apple samples was conducted for each variant before coating for the control sample and after coating for the other variants. The samples were weighed every seven days throughout the 28-day storage period. Weight loss was calculated by subtracting the final weight of the apples from their initial weight. All measurements were recorded using a semi-analytical balance (Radwag S.A., Radom, Poland). Weight loss was calculated using the following formula:

$$U_t = \frac{m_0 - m_t}{m_0} \times 100\%$$

where U_t—percentage weight loss after t days, m_0—initial mass (day 0), and m_t—mass on day t.

3.2. Colour

Colour parameters were measured in two opposite places of three apples (six repetitions) using a Minolta CR-5 colourimeter (Konica Minolta, Tokyo, Japan) in the L^*, a^* and b^* colour space. Measurements were performed with the standard observer 2° and D65 light source.

3.3. Total Soluble Solids

The analysis of the total soluble solids in apples was performed in triplicate using the refractometric method by squeezing the juice from the fruits. The measurement was performed using a refractometer (PAL-3, Atago Instruments, Tokyo, Japan). The results were obtained in °Brix.

3.4. Total Titratable Acidity

To determine the total titratable acidity (TTA) expressed as a percentage of malic acid, 10 mL of apple juice was measured into a beaker. Then, 100 mL of distilled water was added. Using a pipette, 25 mL of this diluted apple juice was transferred to a 50 mL beaker. A magnetic stirrer and electrode were placed in the solution for testing. The titration was carried out by adding a 0.25 mol/L NaOH solution until a pH of 8.1 was reached, which was designated as the neutralisation point. The titratable acidity was calculated in three repetitions using the appropriate formula.

$$TTA = \frac{(100 * V_1 * c)}{V_0}$$

where V_1 = amount of solution used, V_0 = 25 mL and c = 0.25 mol/L NaOH.

3.5. pH

The fruit tissue was cut, ground, and filtered through a sieve to collect the juice. The pH of the fruit juice was measured in three repetitions using a pH meter (SHOTT Instruments, Lab 850, Warsaw, Poland) with three replicates.

3.6. Firmness

The texture of the apples was analysed in nine repetitions using a TA-TX2i texturometer (Stable Micro Systems Ltd., Haslemere, UK) equipped with Texture Expert software (version 2.3), which recorded the force values during the testing process. A penetration test was conducted using a 9 mm pin at a speed of 2 mm/s. Measurements were taken in six replicates, with two punctures made on opposite sides of three apples. The measure of firmness was determined by the maximum force expressed in newtons (N), calculated from the relationship between force and penetration time.

3.7. Respiration Rate

The emissions of ethylene (C_2H_4) and carbon dioxide (CO_2) from the fruits were analysed in duplicates over the course of one hour using the F-950 Analyser (Felix Instruments Inc., Camas, WA, USA). During the analysis, three apples were placed in a sealed 2L jar that was directly connected to the gas analyser, which allowed for an air space of 34 µL for each measurement.

3.8. Sensory Analysis

The sensory analysis of apples was conducted after 7 and 28 days of storage. The apples were washed with water, then cut into pieces and coded accordingly. A sensory evaluation was conducted using a 5-point scale with a group of 40 trained participants, comprising students and staff from the Institute of Food Sciences at Warsaw University of Life Sciences in Poland. Participants were aged 20 to 45. The following qualitative characteristics were assessed: colour, taste, aroma, firmness, and overall fruit acceptability. Excel 2013 software was also used to process the results.

3.9. Statistical Analysis

Statistical analysis of the obtained results was performed using Statistica 13.0 (StatSoft Polska Sp. z o.o., Kraków, Poland), utilising one-way ANOVA, Pearson's correlation and Tukey's HSD post hoc test at a significance level of 0.05. Excel 2013 software was also used to process the results. Error bars represent standard deviation (SD).

4. Results and Discussion

4.1. The Effect of Phenolic Acids on the Rheological Properties of Coating Solutions

Rheological properties concern the study of material flow and deformation under stress, which enables better optimisation of processing conditions and desired product attributes [48]. The flow properties of coating solutions enable the analysis of factors related to spreadability, thickness, homogeneity, mechanical properties, microstructure, and application methods [38,49]. The flow (Figure 1) and viscosity curves (Figure 2) of the prepared coating solution, based on apple pectin and its mixtures with phenolic acids (caffeic and protocatechuic), revealed differences in the rheological behaviour of the tested samples. All samples demonstrated non-Newtonian, shear-thinning flow behaviour, which is consistent with previous observations of pectin solutions [50,51].

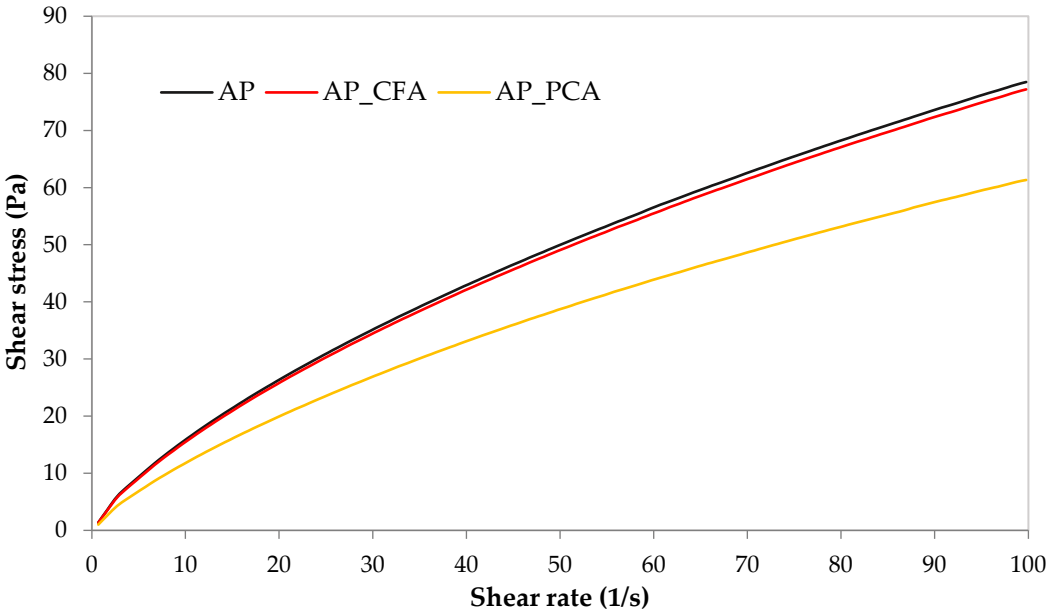


Figure 1. Flow curves of coating-forming solutions based on apple pectin (AP) with the addition of caffeic acid (AP_CFA) and protocatechuic acid (AP_PCA).

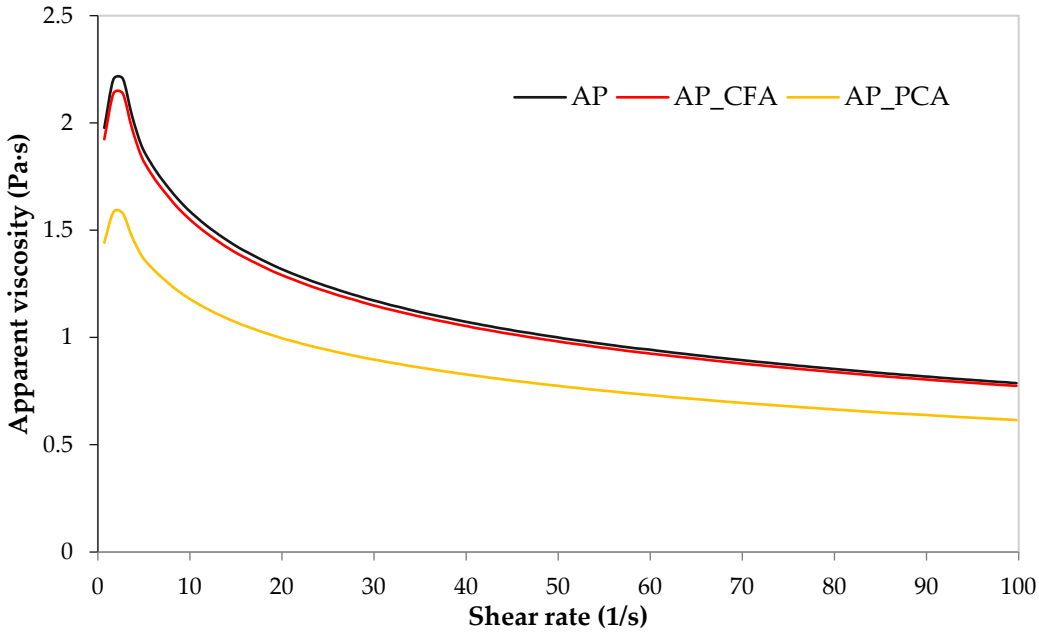


Figure 2. Viscosity curves of coating solution based on apple pectin (AP) with the addition of caffeic acid (AP_CFA) and protocatechuic (AP_PCA) acid.

The addition of protocatechuic acid led to a decrease in apparent viscosity across all levels of applied shear stress. This effect may be related to changes in the molecular structure of pectin, particularly how protocatechuic acid influences the dissociation degree of carboxyl groups and potentially disrupts the network formation between polysaccharide chains. These findings align with those reported by Karaki et al. [52,53], who demonstrated that the addition of phenolic acids, such as ferulic acid, reduces molecular packing density and intermolecular interactions in pectin systems, resulting in decreased viscosity. In the case of caffeic acid, the viscosity remained unchanged compared to the control sample (coating solution without phenolic acids). This may be attributed to weaker interactions of this acid with pectin chains and a lower capacity to form hydrogen bonds. The differences

in rheological behaviour can also be interpreted in terms of changes in the hydrophobicity of pectin molecules after adding phenolic compounds. According to Zhang et al. [54], modifying carboxyl side groups and rhamnose content is crucial for the flow properties of apple pectin. The presence of protocatechuic acid may disrupt the structure and length of pectin aggregates, thereby altering viscosity.

4.2. Release Kinetics of Caffeic and Protocatechuic Acids from Pectin Films

Active packaging is a method used for food preservation that helps maintain the quality, safety, and integrity of food throughout its shelf life [55]. According to Regulation (EC) No. 450 [56], active packaging refers to packaging systems that interact with food to deliberately incorporate components that either release or absorb substances into the packaged food or its surrounding environment. The inclusion of bioactive substances can improve the ability of the packaging material to preserve the physicochemical properties of food, thereby extending its shelf life [57,58]. Some types of packaging films utilise materials that are sensitive to oxygen or contain antioxidants. To achieve a controlled or targeted release of bioactive compounds with specific functional properties, it is essential to consider various factors, including the type of bioactive compound, its delivery method, and the environmental conditions present [59]. In packaging films, parameters such as release rate, diffusion coefficient, and cumulative release are utilised [60]. There are two primary mass transport mechanisms that regulate the release of molecules: internal (diffusion within the material) and external (transport from the material to the surrounding atmosphere or food). Besides facilitating the mass transport of active substances, diffusion can also lead to swelling and/or plasticization of the polymer, which may disrupt the release process [61].

The release kinetics of caffeic and protocatechuic acids from pectin films in a 96% ethanol medium are illustrated in Figure 3. The analysed films displayed excellent solubility in water, as discussed in our previous work [36]. Consequently, distilled water or a 50% ethanol solution did not show suitable release kinetics due to immediate dissolution. The different behaviours of the phenolic acids in ethanol are likely attributed to the varying chemical compositions and properties of the acids. Additionally, the release process may be affected by interactions among the components, specifically pectin, acid, glycerol, and ethanol. Pearson's linear correlation coefficients for both acids were notably high, with protocatechuic acid exhibiting a higher value of 0.9733. Furthermore, all tested variants showed linear correlation coefficients close to unity, indicating strong linear relationships between the variables.

Hernández-García, Vargas, and Chiralt [62] performed methanolic extraction of ferulic, p-coumaric, and protocatechuic phenolic acids from PLA-PHBV blend films. Subsequently, spectrophotometric determinations yielded similar values (83.0 $\pm$ 0.03, 80.0 $\pm$ 0.1, and 79.0 $\pm$ 0.04 g of retained compound/100 g of incorporated compound, respectively). After conducting kinetic studies on the release of active substances into food simulants, it was found that phenolic acids behaved differently in both simulants (10% v/v aqueous ethanol solution and 50% v/v aqueous ethanol solution). This difference depended on the chemical affinity of the compounds for the polymer matrix and their solubility. Furthermore, disintegration occurred more rapidly for films containing acids compared to acid-free polyester film, which remained intact for 20 min in contact with both types of simulants. This confirms that the release of phenolic acid and the subsequent pH drop in the aqueous environment significantly influence film disintegration. The highest initial release rate and equilibrium release coefficient were achieved for ferulic acid in both simulants, while protocatechuic acid exhibited the slowest release from films in both simulants. It was also observed that the maximum amount of released acids was significantly lower compared to the solubility of the compounds in water, which increases with the ethanol content in

the simulants. Differences in kinetic parameters are attributed to variations in chemical interactions between the compounds and the polyester matrix, as well as differing degrees of relaxation. The release rate and equilibrium release amount of all phenolic acids were higher in the simulant with a greater proportion of ethanol. Benbettaïeb et al. [63] studied the release kinetics of ferulic acid and tyrosol from chitosan-gelatin-based films, both non-irradiated and irradiated at a dose of 60 kGy, in an aqueous environment with a pH of 7 or lower. The non-irradiated films contained a significantly higher amount of ferulic acid, likely due to ferulic acid's greater ability to interact with the polymer network or promote cross-linking. This interaction leads to improved mechanical properties and permeability of the films. However, there is limited research focusing on the antioxidant activities of phenolic acids and their relationship to the mechanisms of their release.

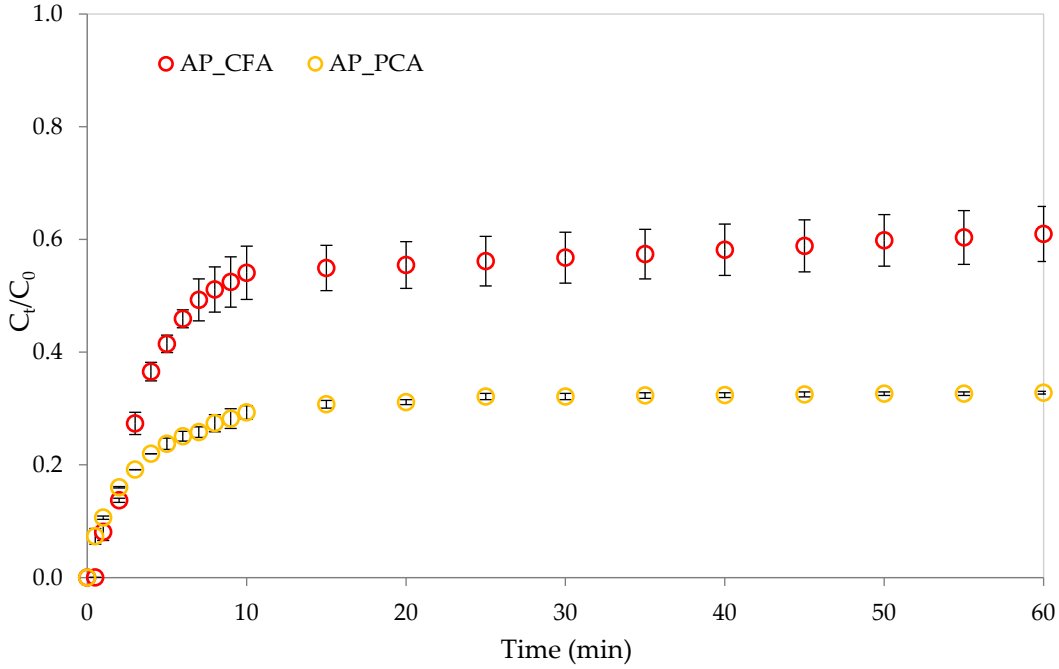


Figure 3. Release kinetics of caffeic acid (CFA) and protocatechuic acid (PCA) from apple pectin (AP) packaging films into aqueous medium (96% ethanol). (C_t is the antioxidant concentration in the medium at time t; C_0 is the initial antioxidant concentration in films prior to release).

Controlled-release packaging provides several advantages over traditional methods for extending the shelf life of food. This technology enables the adjustment of antioxidant activity according to the food's storage conditions within the package, ensuring maximum freshness [59]. Typically, the active ingredient can be introduced through methods such as adsorption, covalent immobilisation on the polymer surface, or direct incorporation into the polymer structure. When controlled-release technology involves entrapment within a polymer matrix, the film thickness is critical, as it significantly influences the diffusion characteristics of the active ingredient [60]. Generally, this means that the release rate will decrease as the film thickness increases. Moreover, when nanofillers are added, there is a reduction in the release rate due to an increased diffusion path length, which may result from a decrease in polymer flexibility and film elasticity [64].

4.3. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the Weight Loss of Apples During Storage

The loss of mass in fruit during storage significantly affects its external appearance, which is a key factor in determining quality [65]. Thus, a primary objective of applying edible coatings to fresh fruit is to minimise this loss of mass during storage [1]. Figure 4 shows

the weight losses of uncoated and coated apples with pectin, both with and without caffeic or protocatechuic acids, over a 28-day storage period. It is evident that uncoated apples experienced the highest weight loss, ranging from 2.46 ± 0.2% at 7 days to 8.23 ± 0.06% at 28 days. This trend indicates that the coating effectively reduced moisture loss caused by evaporation. Considering the effects of bioactive coatings, there was not much difference between the coatings with and without the addition of phenolic acid. The weight loss values ranged from 1.77% to 1.81% at 7 days, and from 4.67% to 4.82% at 28 days. Moreover, the highest weight losses for all samples were observed up to 21 days, after which there were lower differences noted between days 21 and 28. The rapid loss of water results in the drying and degradation of fruit [66]. Studies have shown that the application of edible coatings, whether made from apple pectin alone or mixed with phenolic acids, significantly reduces the mass loss of apples during storage over subsequent weeks. In contrast, uncoated apples do not show this behaviour. Therefore, it can be concluded that developed edible coatings serve as a barrier, preventing moisture loss from the fruit's surface and helping to maintain its quality during storage. Kassebi et al. [17] noted a higher value (approximately 17.5%) of weight loss of uncoated Golden Delicious apples stored at room temperature (24 ± 1 °C) and 60% air humidity for 4 weeks. The average weight reduction in apples was 5.29% in the first week of measurement and 25.32% at the end of storage. The authors suggested that increased weight loss indicates the internal transpiration of the fruit, mainly due to moisture loss. In general, changes in the fruit during ripening are related to an increase in ethylene synthesis, the respiration process, and the process of pectin degradation that plays an essential role in cell wall degradation, affecting shelf life. However, the mechanisms of these processes are related to many factors, including fruit variety, harvesting time, and storage conditions (temperature and relative humidity). In the case of coated apples, the coating acts as a permeable membrane, which affects biological processes such as respiration and the migration of water vapour [67]. Therefore, edible coatings significantly reduce apple weight loss by forming a barrier that controls moisture and gas exchange, and they inhibit microbial growth by reducing water activity and oxygen availability, thereby extending shelf life and maintaining quality [68].

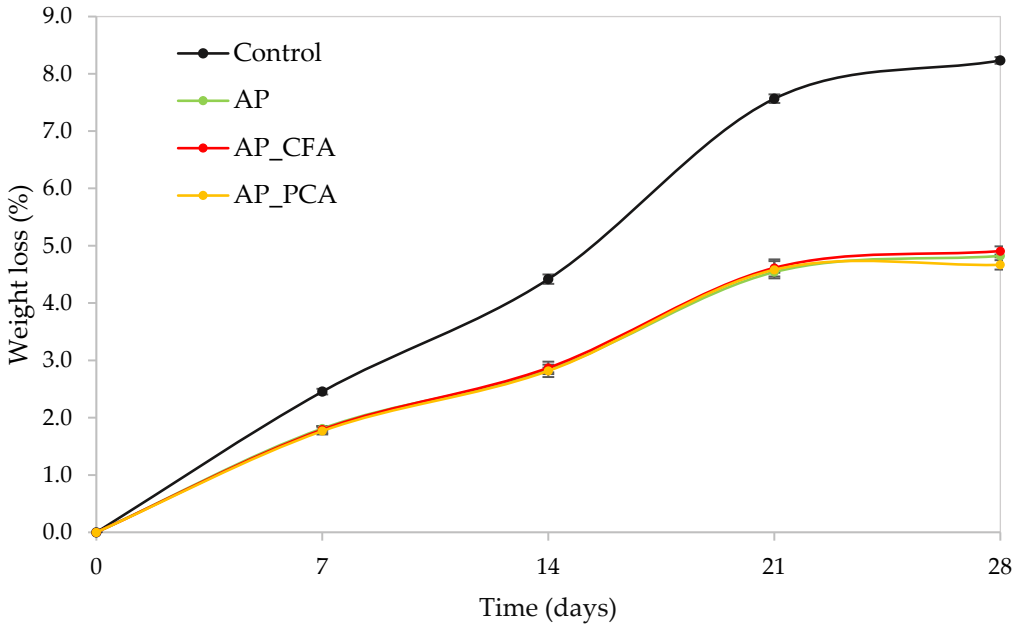


Figure 4. Weight loss of uncoated (Control) and coated apple with the coating solution based on apple (AP) incorporated with caffeic (AP_CFA) and protocatechuic (AP_PCA).

Storing fruit at room temperature may lead to reduced juice content due to increased moisture loss [69]. Studies by Trebar, Žalik, and Vidrih [70] demonstrate that temperature has a significant effect on weight loss in apples. Notable differences arise when uncoated Golden Delicious apples are stored at ambient temperature (approximately 25 °C) compared to the recommended storage temperature of 5 °C. Due to the high water content in the fruit, which is about 80–90% by weight, rapid evaporation can lead to spoilage and a reduced shelf life. Additionally, the natural barrier on the fruit, along with the use of an appropriate type and amount of edible coating, can influence the extent of weight reduction [71]. Even a small loss of 3 to 10% can negatively impact the appearance of apples, resulting in fruit shrinkage and a decline in their visual appeal [72]. Fruits become unsuitable for sale if their weight loss results in visual changes that are unacceptable to consumers [73]. A 59% reduction in weight loss was observed in apples of the Golab Kohanz variety when they were coated with a nanochitosan solution containing a 0.5% chitosan concentration during the entire storage period. These findings demonstrate the potential of using nanochitosan coatings for climacteric fruits, thereby extending their shelf life [74]. Apples coated with potato starch and polyvinyl alcohol demonstrated better weight retention during storage compared to uncoated apples [75]. Furthermore, coating Golden Delicious apples with water walnut starch-based coatings infused with rosemary essential oil (REO) at concentrations of 0.10%, 0.25%, and 0.50% (w/v) positively influenced their post-harvest stability during 120 days of cold storage. The control apples, which were not coated, experienced the highest weight loss, which gradually increased throughout the storage period, in contrast to the coated apples [10]. Maintaining moisture in apples enhances their appearance, preserves firmness, and increases marketable weight, positively impacting growers' income [65]. Jahanshahi et al. [76] demonstrated the potential of using tragacanth gum as a coating to improve postharvest shelf life and maintain the quality of apple fruits, thereby reducing postharvest losses. The results showed that, in both Red Delicious and Golden Delicious cultivars, the shelf life was significantly increased by applying a coating composed of 10 kg/m^3 of gum. This was evidenced by a reduced decrease in firmness and considerably reduced fruit weight loss during 120 days of storage at low temperature (0 ± 1 °C) and 85–95% relative humidity. The study suggests minimising postharvest losses by using an appropriate edible coating.

4.4. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the Colour of Apples During Storage

In the view of consumers, one of the most important quality features of apples is their colour, which is influenced by cultural preferences and consumer choices. When cultivating apples, factors such as the consistency of colour, the intensity of blush (if applicable), as well as the presence of dents or bruises and the stage of maturity are often considered. Additionally, after harvesting, colour remains a significant quality attribute, affected by storage conditions, which can greatly influence the final quality of the fruit [77,78]. Enzymatic browning in fresh fruit causes skin and flesh discolouration, which reduces the product's commercial value. This browning is highly noticeable to consumers, influencing their perception of freshness and significantly affecting their purchasing decisions [14]. Recent studies indicate that coatings effectively regulate the critical physiological and biochemical changes that lead to enzymatic browning [66]. Figure 5 illustrates both the external and internal appearances of the analysed uncoated and coated apples during storage. It can be observed that the coated apples appear greener compared to the control samples, which are more yellow. There is no visible difference between the samples coated with formulations containing or lacking phenolic acids, indicating that the apple pectin coating serves as a protective layer during storage, regardless of the presence or absence of active compounds.

However, the internal tissue of the uncoated apples appears lighter, while the coated apples show a rapid onset of enzymatic browning compared to the controls.

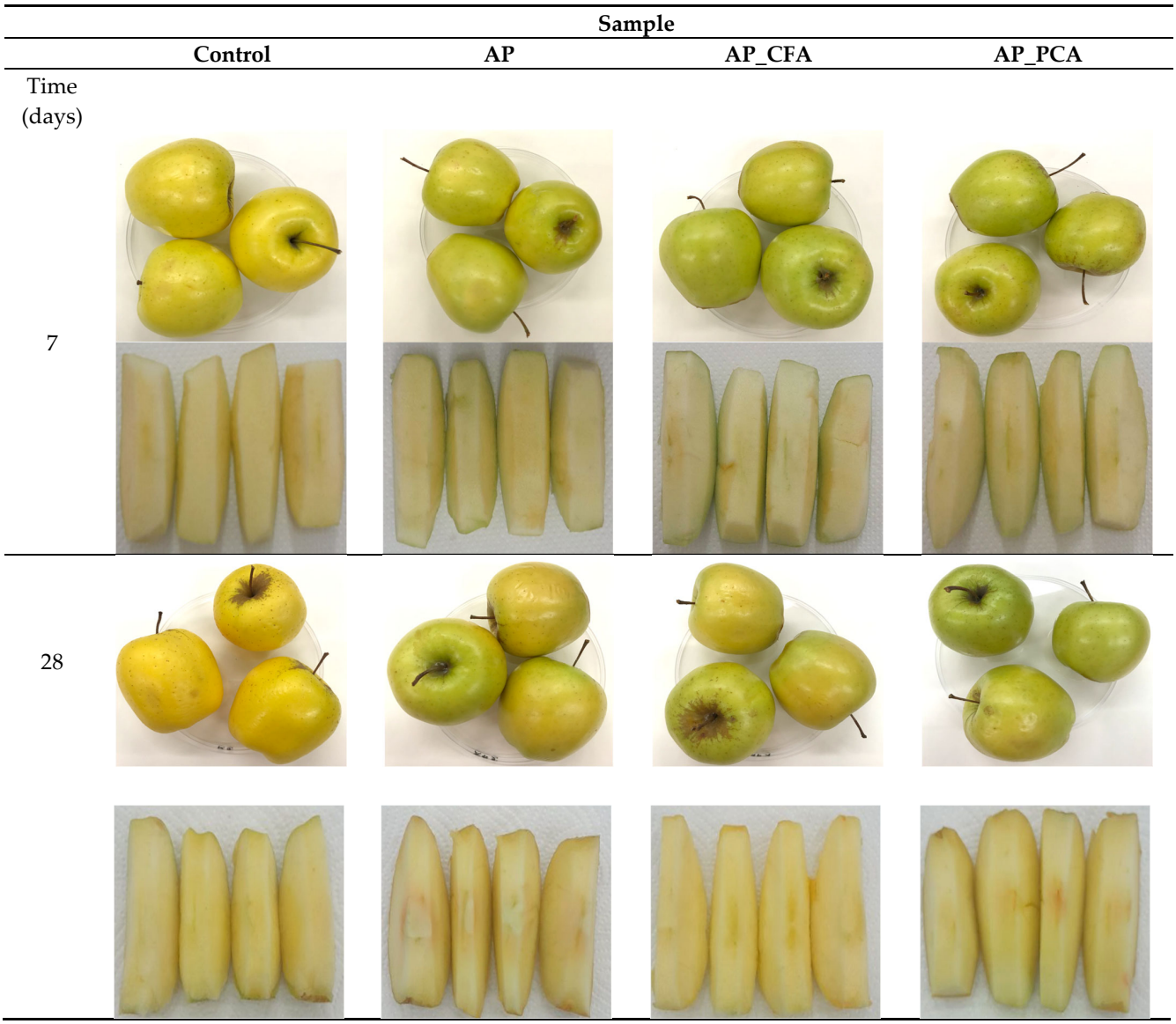


Figure 5. The appearance of uncoated (Control) and coated apples with the coating solution based on apple (AP) incorporated with caffeic (AP_CFA) and protocatechuic (AP_PCA) at 7 and 28 days of storage.

The yellow colour of Golden Delicious apples comes from pigments in their skin, which mainly consist of flavonoids, particularly flavonols and flavones, as well as carotenoids. Meanwhile, chlorophyll is responsible for the green colour of the skin [79]. The colour change from green to light green and then to yellow is likely the result of pigment degradation during storage. This process is influenced by the synthesis of carotenoids and the decomposition of chlorophyll. Moreover, these changes are affected by the activity of polyphenol oxidase, the most common enzyme involved in the browning process of apple flesh [17]. Chlorophyll synthesis is a continuous process that occurs before fruit ripening, but it gradually decreases over time. This decline initially leads to a reduction in the green colour of the fruit. Eventually, chlorophyll is replaced by other pigments, resulting in the appearance of visible yellow shades [80].

The results for the colour parameters L^*, a^*, and b^* for the evaluated apples are presented in Table 2. In the CIELAB colour space, L^* represents lightness; the a^* parameter indicates colour on a red-green scale, where positive values reflect red and negative values denote green. The b^* parameter indicates colour on a yellow-blue scale, with positive values corresponding to yellow and negative values to blue. A high L^* value indicates that the fruit has a light skin colour. After 28 days of storage, the colour of uncoated apples remained stable, although some changes in the colour components were observed throughout their shelf life.

Table 2. The L^*, a^*, and b^* colour parameters of uncoated (Control) and coated apples with the coating solution based on apple (AP) incorporated with caffeic (AP_CFA) and protocatechuic (AP_PCA).

Time (Days)	Sample			
	Control	**AP**	**AP_CFA**	**AP_PCA**
		L*		
0	$75.77 \pm 1.07^{a,A}$			
7	$75.40 \pm 2.45^{a,A}$	$72.03 \pm 5.56^{a,A}$	$72.05 \pm 1.26^{b,A}$	$72.21 \pm 1.76^{a,A}$
14	$75.01 \pm 1.98^{a,B}$	$70.45 \pm 1.50^{a,A}$	$69.49 \pm 1.40^{a,A}$	$68.64 \pm 2.12^{a,A}$
21	$74.70 \pm 1.59^{c,B}$	$70.30 \pm 1.50^{a,A}$	$68.79 \pm 2.44^{a,A}$	$68.01 \pm 1.87^{a,A}$
28	$74.67 \pm 1.79^{a,B}$	$64.64 \pm 7.31^{a,A}$	$67.37 \pm 2.46^{a,A}$	$64.66 \pm 7.17^{a,A}$
		a*		
0	$-11.29 \pm 0.68^{a,A}$			
7	$-7.81 \pm 2.32^{b,A}$	$-9.18 \pm 0.94^{a,A}$	$-9.26 \pm 1.20^{a,A}$	$-8.11 \pm 1.49^{a,A}$
14	$-4.64 \pm 2.00^{b,B}$	$-8.57 \pm 1.38^{a,A}$	$-8.51 \pm 1.35^{a,A}$	$-8.04 \pm 1.54^{a,A}$
21	$-2.75 \pm 2.16^{b,B}$	$-6.12 \pm 1.60^{b,A}$	$-5.96 \pm 2.09^{b,A}$	$-5.58 \pm 1.91^{b,A}$
28	$-2.16 \pm 1.28^{b,A}$	$-4.35 \pm 1.38^{b,A}$	$-3.72 \pm 1.64^{b,A}$	$-3.96 \pm 1.65^{b,A}$
		b*		
0	$46.83 \pm 0.63^{a,A}$			
7	$55.21 \pm 2.33^{b,B}$	$46.29 \pm 2.74^{a,A}$	$46.28 \pm 1.73^{a,A}$	$46.13 \pm 1.93^{a,A}$
14	$56.18 \pm 1.47^{b,B}$	$44.51 \pm 1.68^{a,A}$	$43.68 \pm 1.41^{a,A}$	$44.15 \pm 2.14^{a,A}$
21	$57.22 \pm 2.43^{b,B}$	$44.39 \pm 2.26^{a,A}$	$43.39 \pm 2.04^{a,A}$	$43.93 \pm 2.03^{a,A}$
28	$57.76 \pm 3.08^{b,B}$	$42.97 \pm 4.25^{a,A}$	$42.69 \pm 2.64^{a,A}$	$42.59 \pm 1.43^{a,A}$

Mean values ± standard deviations. Different superscript letters ($^{a–c}$) within the same column or rows (^{A,B}) indicate significant differences between the samples ($p < 0.05$).

An analysis of the L^* parameter revealed that it fluctuated between 64.64 ± 7.31 and 72.21 ± 1.76 for coated apples, while uncoated apples ranged from 74.67 ± 1.79 to 75.77 ± 1.07 (Table 2). This suggests that time had a significant impact on changes in lightness. The decrease in lightness observed on the 28th day of storage for apples coated with a pectin solution containing added caffeic acid and protocatechuic acid was attributed to the development of brown spots on the apples' surface. A decrease in the L^* parameter was observed, which corresponds to a reduction in lightness as the skin surface yellowed and darkened due to spoilage. The results showed the significant differences between the control samples and the coated apples after 14 days of storage. There were no significant differences among the types of coatings used, suggesting that all pectin coatings, with and without the phenolic acids, were effective in the reduction in lightness ($p < 0.05$). This change may be attributable to various alterations in the fruit's tissues, including enzymatic browning, dehydration, and ripening. Additionally, the colour change in apples is linked to weight loss, which affects ethylene production and respiration. Notably, the smallest reduction in lightness values was observed in the control group of uncoated apples. Kassebi et al. [17] investigated the changes in colour of Golden Delicious apples over a six-week storage period at 24 °C and 60% relative humidity. They found that the L^*

parameter also decreased slightly, from 74.81 to 71.27, as storage time increased from 1 to 6 weeks.

The a^* and b^* colour parameters are closely related and indicate the ripening process of fruit. As fruit ripens, the amount of green colour (indicated by negative a^*) decreases, while the amount of yellow colour (indicated by positive b^*) increases. This colour change occurs due to the decomposition of chlorophyll, which leads to the synthesis of other pigments, primarily anthocyanins or carotenoids. The most noticeable change is in the colour of the skin, which typically transitions from green to a lighter shade. The brown-green colour observed in apples can be attributed to the degradation of chlorophyll, particularly the loss of the Mg^{2+} ion from chlorophyll, resulting in the formation of phaeophytin. Several factors can influence chlorophyll degradation, including exposure to heat, light, and oxidising agents. Research indicates that the phaeophytin reaction occurs more rapidly in the presence of heat due to protein denaturation [81]. In conclusion, using edible coatings that contain phenolic acids may help reduce chlorophyll degradation and preserve the green colour of Golden Delicious apples.

Negative a^* parameter values were observed for all samples (Table 2), indicating a green skin colour, which is characteristic of the Golden Delicious cultivar. The highest increase in parameter a^*, from -11.29 ± 0.68 to 2.16 ± 1.28, was observed for uncoated apples, indicating their advanced ripeness compared to the other samples. Pectin coatings mitigated this increase, resulting in values of -4.35 ± 1.38 for coated samples without phenolic acids, -4.72 ± 1.64 for those with caffeic acid, and -3.96 ± 1.65 for those with protocatechuic acid. It is observed that all bioactive coatings effectively affected the lower values of parameter a^* after 14 and 21 days of storage. However, among the types of coatings used, there were no significant differences in the values ($p < 0.05$). This is probably attributed to the lack of effect of phenolic acids on this parameter for pectin films.

The b^* parameter values for control apples ranged from 46.83 ± 0.63 to 57.76 ± 3.08, demonstrating an increasing trend with each week of storage (Table 2). In contrast, the b^* colour parameter for coated apples decreased as storage time increased. All b^* values remained positive, indicating that the apples consistently fell within the yellow colour range throughout each storage period. Storing the apples for an additional two months did not significantly change the fruit's colour; however, slight differences were noted across all colour quality classes. As weight loss increased, the b^* value also increased, suggesting that the apples became more yellow. This water loss may lead to a concentration of pigments in the apples, enhancing the yellow colour. Time had an impact on colour change in all combinations, but the application of coatings helped limit this change. The experiment confirmed that using coatings delays the yellowing of apple skin. This effect was observed for each coating formulation and at all times tested, indicating that all coated apples were significantly affected by bioactive coatings in a similar manner ($p < 0.05$). There were no differences between the types of coatings used.

Coatings with added antioxidants, such as ascorbic acid, calcium chloride, or calcium lactate, are particularly effective at maintaining colour, mostly in fresh-cut apples. Incorporating antimicrobial agents, such as chitosan or essential oils, significantly reduces the overall microbial load, both for minimally processed and non-processed fruits [82].

4.5. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the Total Soluble Solids of Apples During Storage

Both coated and uncoated apples showed an increase in total soluble solids when comparing storage periods of 7 and 28 days (Table 3). For control samples, the values increased from 13.47 ± 0.37 to 14.95 ± 0.48 °Brix at 28 days of storage. In contrast, for the coated sample, the increase in this parameter was within the range of 13.52–13.57 °Brix. Thus, the coating process significantly decreased the rate of increase in the total soluble

solids in fruit throughout the period, as only a slight increase in extract was observed in the coated samples after 28 days of storage. The highest extract content was recorded in the control sample on day 28 of storage, measuring 14.95 ± 0.48 °Brix. These results indicate that, from a physicochemical perspective, significant changes occurred during the storage of Golden Delicious apples, depending on their state of ripeness. Kassebi et al. [17] also observed an increase in extract during the storage of the same variety of apples. The initial extract value was 13.61 °Brix in the first week of storage at room temperature, which increased to 14.78 °Brix after six weeks. This increase may be attributed to a decrease in fruit moisture, leading to a higher concentration of sugars within the fruit, the breakdown of starch into sugars, or the hydrolysis of cell wall polysaccharides. Regarding the type of coating, there were similar tendencies among the samples. A significant increase in total soluble solids was observed in control samples after 21 and 28 days of storage ($p < 0.05$).

Table 3. Total soluble solids, titratable acidity and pH of uncoated (Control) and coated apples with the coating solution based on apple (AP) incorporated with caffeic (AP_CFA) and protocatechuic (AP_PCA).

Time (Days)	Sample			
	Control	AP	AP_CFA	AP_PCA
	TSS (°Brix)			
0	13.47 ± 0.37 ^{a,A}			
7	13.77 ± 0.11 ^{a,A}	13.50 ± 0.10 ^{a,A}	13.48 ± 0.23 ^{a,A}	13.53 ± 0.40 ^{a,A}
14	14.03 ± 0.39 ^{a,A}	13.50 ± 0.24 ^{a,A}	13.52 ± 0.21 ^{a,A}	13.50 ± 0.37 ^{a,A}
21	14.35 ± 0.19 ^{b,B}	13.53 ± 0.07 ^{a,A}	13.52 ± 0.18 ^{a,A}	13.55 ± 0.36 ^{a,A}
28	14.95 ± 0.48 ^{b,B}	13.53 ± 0.19 ^{a,A}	13.52 ± 0.32 ^{a,A}	13.57 ± 0.27 ^{a,A}
	Titratable acidity (% of malic acid)			
0	1.37 ± 0.02 ^{b,A}			
7	0.82 ± 0.05 ^{a,A}	0.93 ± 0.05 ^{a,A}	0.91 ± 0.02 ^{a,A}	0.89 ± 0.11 ^{a,A}
14	0.90 ± 0.07 ^{a,B}	0.79 ± 0.08 ^{a,AB}	0.77 ± 0.03 ^{a,A}	0.77 ± 0.09 ^{a,A}
21	0.88 ± 0.04 ^{a,B}	0.75 ± 0.05 ^{a,A}	0.76 ± 0.08 ^{a,A}	0.77 ± 0.05 ^{a,A}
28	0.80 ± 0.05 ^{a,A}	0.79 ± 0.05 ^{a,A}	0.76 ± 0.05 ^{a,A}	0.73 ± 0.02 ^{a,A}
	pH			
0	3.59 ± 0.04 ^{a,A}			
7	3.84 ± 0.02 ^{b,A}	3.84 ± 0.01 ^{b,A}	3.83 ± 0.03 ^{a,A}	3.79 ± 0.05 ^{a,A}
14	3.90 ± 0.03 ^{b,A}	3.98 ± 0.09 ^{b,A}	3.98 ± 0.06 ^{b,A}	4.04 ± 0.01 ^{b,A}
21	4.00 ± 0.02 ^{b,B}	4.09 ± 0.06 ^{b,A}	4.12 ± 0.08 ^{b,A}	4.16 ± 0.01 ^{b,A}
28	4.05 ± 0.06 ^{b,A}	4.11 ± 0.07 ^{b,A}	4.12 ± 0.03 ^{b,A}	4.26 ± 0.04 ^{b,A}

Mean values ± standard deviations. Different superscript letters (^{a,b}) within the same column or rows (^{A,B}) indicate significant differences between the samples ($p < 0.05$).

4.6. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the Acidity of Apples During Storage

Acidity in fruits is determined by the amount of organic acids they contain, which significantly impacts both their physiological metabolism and sensory qualities. Additionally, acidity tends to decrease over time during storage and ripening. This decline is due to enzymatic processes that occur, leading to an increase in the fruit's sweetness [10]. Understanding how the storage process affects acidity is crucial for improving the quality of fruit after harvest. Fruit acidity is primarily determined by the presence of one or two acids, such as citric, tartaric, and malic, which contribute to the sour taste. Conversely, the sweet taste of apples depends on the amount of soluble solids. For apples, approximately 90% of all organic acids consist of malic acid, at a concentration ranging from 1.72 to 29.27 mg/g. During apple growth, malate content increases; however, it decreases during ripening and storage. Citric acid, present in small amounts, also contributes to the acceptability of apples.

Conventional storage of apples at 0 ± 0.1 °C has been found to be ineffective in maintaining fruit acidity, an essential quality parameter [83].

It was observed that the acidity of uncoated apples decreased the most significantly (Table 3), from 1.37 ± 0.02 to 0.80 ± 0.05%, similar to apples coated with the formulation without phenolic acids (0.79 ± 0.05%). The lowest values of acidity were observed for the sample coated with solutions containing caffeic and protocatechuic acids, 0.76 ± 0.05 and 0.73 ± 0.02%, respectively. After analysing the obtained results, it can be concluded that coated apples exhibited the most significant change in malic acid content during storage, leading to a considerable alteration in their taste. This phenomenon may arise from the fact that, during storage, the starch content decreases while the sugar content increases, resulting in a decrease in the acidity of the apples [84]. However, considering the coating type, similar tendencies were observed among the samples. A significant increase in titratable acidity was observed in uncoated apples after 14 and 21 days of storage ($p < 0.05$). In a study conducted by Rashid et al. [85], apples were coated with solutions containing fenugreek and linseed, then stored at 25 ± 2 °C for 35 days. The experimental results indicated that the titratable acidity values decreased for both coated and uncoated fruits. This reduction in acidity can likely be attributed to the coatings, which slowed down the respiration process and, consequently, the metabolic processes, including acid decomposition. It may be related to a reduction in gas exchange with the external environment [86].

4.7. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the pH of Apples During Storage

The occurrence of an increase in the pH of fruits during storage is related to their respiration, during which the oxidation of organic acids occurs and the decrease in acids present in the fruits with the passage of time [87]. The pH of the fruit systematically increased in all samples over 28 days of storage (Table 3). No significant fluctuations were observed between combinations regarding storage time, and all samples exhibited similar pH values, ranging from 3.59 to 4.26. The values slightly increased from 3.59 ± 0.04 to 4.05 ± 0.06 for control samples. Coated apples showed higher values: 4.11 ± 0.07 for samples coated with the solution without phenolic acids, and 4.12 ± 0.03 and 4.26 ± 0.04 for samples coated with solutions containing caffeic and protocatechuic acids, respectively. However, after 21 and 28 days of storage, a significant increase in pH was observed for uncoated apples (4.00 ± 0.02) and apples coated with a solution containing protocatechuic acid (4.26 ± 0.04), respectively ($p < 0.05$). An increase in pH in apples during storage is a natural part of the ripening process, where apples become less acidic and more alkaline as organic acids, like malic acid, decrease due to respiration [88]. Coated apples were less ripe; however, their pH values were higher than those of the controls. This may suggest that the increase in pH is related to changes in the fruits' chemical composition. In contrast, Vieira et al. [89] reported a much higher pH of 4.97 ± 0.04 for fresh Golden Delicious apples, while Alegre et al. [90] found an apple pH of 4.16. Variations in pH can be attributed to plant growth conditions. During storage, organic acids in fruits act as respiratory substrates and are slowly utilised, which increases pH and decreases acidity [4]. Moreover, pH may be related to increased dry matter content and polysaccharide depolymerisation with extended storage [85].

4.8. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the Firmness of Apples During Storage

The primary challenge in apple storage is maintaining firmness, which significantly influences consumer sensory perception and commercial value. The loss of firmness, or softening, is an undesirable process during apple ripening. Higher fruit hardness and

firmness correlate with increased juiciness, crunchiness, and reduced mealiness, leading to greater consumer acceptance [91]. The firmness results, presented in Table 4, ranged from 58.03 ± 6.50 to 36.84 ± 1.30 N for fresh apples and after 28 days of storage. For the coated apples, a lower rate of decrease in values was observed, ranging from 43.64 to 44.61 N, with minimal differences among the types of coatings. Taking into account the coating type, no significant effect was observed between formulations, indicating that a significant reduction in firmness was achieved for uncoated apples after 14, 21, and 28 days of storage ($p < 0.05$) compared to coated samples. In general, apples characterised by greater hardness are more resistant to mechanical damage and are more suitable for longer storage [92]. Softer fruit, due to enzymatically induced modifications of polysaccharide cell walls and their networks, is more susceptible to fungal infections [93]. In contrast, the loss of firmness and softening of apples are primarily attributed to modifications in pectin within the cell walls, with minor changes in hemicellulose and cellulose, as well as cellulase activity [94]. Edible coatings limit the action of enzymes responsible for pectin decomposition, thereby slowing metabolic processes and maintaining apple firmness [85]. Polysaccharide-based coatings are used as structural reinforcement to ensure cell wall integrity and mitigate mechanical damage that may occur during transport, thereby ensuring appropriate hardness and texture [5]. The hardness of Golden Delicious apples may also be influenced by soil and climatic conditions [70].

Table 4. Firmness of uncoated (Control) and coated apples with the coating solution based on apple (AP) incorporated with caffeic (AP_CFA) and protocatechuic (AP_PCA).

Time (Days)	Sample			
	Control	AP	AP_CFA	AP_PCA
	Firmness (N)			
0	58.03 ± 6.50 ^{c,A}			
7	50.77 ± 2.40 ^{b,B}	49.01 ± 1.60 ^{b,AB}	48.32 ± 2.60 ^{a,A}	48.03 ± 1.60 ^{a,A}
14	42.80 ± 3.70 ^{a,A}	45.86 ± 2.70 ^{b,AB}	47.40 ± 2.60 ^{a,B}	46.45 ± 2.80 ^{a,B}
21	39.73 ± 2.50 ^{a,A}	43.94 ± 3.70 ^{a,B}	44.70 ± 3.30 ^{a,B}	45.33 ± 2.90 ^{a,B}
28	36.84 ± 1.30 ^{a,A}	43.64 ± 3.50 ^{a,B}	44.20 ± 4.10 ^{a,B}	44.61 ± 4.80 ^{a,B}

Mean values ± standard deviations. Different superscript letters ($^{a–c}$) within the same column or rows (^{A,B}) indicate significant differences between the samples ($p < 0.05$).

One method of limiting the loss of apple firmness is to use an appropriate concentration of coating containing calcium and wax combined with hydrocooling, which delays fruit ripening and maintains quality during long-term storage [95]. It was observed that using 2% potato starch and 2% apricot kernel oil maintained higher apple hardness, measuring 62.2 N and 62.8 N, respectively, during storage at room temperature and in a cold store [96]. The loss of firmness and limitation of internal browning of Granny Smith apples were also obtained by covering the apples with a wax coating [97]. On the other hand, the factor contributing to fruit softening is the production and accumulation of ethylene, which may induce undesirable physiological and biochemical changes in fruits, including the expansion of cell walls and the activation of proteolytic and pectolytic enzymes [98]. If the hardness drops below 44 N, it is considered to be the beginning of the occurrence of flouriness [99].

4.9. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the Respiration of Apples During Storage

Fruit ripening processes involve the exchange of gases, such as carbon dioxide and oxygen, with the external atmosphere during storage. To maintain the appropriate quality of apples during storage, reducing ethylene metabolism and fruit respiration is neces-

sary [100]. Apple skin is covered with a complex mixture of lipids that protects the fruit from environmental stress and affects gas exchange. The hydrophobic layer allows controlled diffusion of oxygen and carbon dioxide, facilitating proper fruit respiration and preventing the accumulation of carbon dioxide and ethylene. This layer is essential for maintaining the freshness and quality of apples during storage [101]. Unfortunately, cuticular wax can change during ripening and storage, increasing the content of alcohols, esters, and fatty acids in its composition, affecting the formation of a greasy or oily layer on the fruit's surface. The increase in skin greasiness after harvesting and storage at room temperature decreases their commercial value. Additionally, as apples ripen, increased ethylene production affects the fruit's physiological and metabolic functions, supporting ripening and the synthesis and degradation of cuticular wax. Due to increased wax synthesis with ethylene, the fruit skin becomes oily [102]. Reaching an oxygen level below 8% in the fruit limits endogenous ethylene production and extends its shelf life [66]. Lower oxygen content can also lead to the initiation of anaerobic respiration, causing ethanol accumulation and the development of undesirable flavours, which negatively affect the sensory experience of consumers [103]. Recent reports indicate that various types of edible coatings inhibit the exchange of carbon dioxide, oxygen, and ethylene, effectively delaying ripening and maintaining quality after harvest [66]. As noted from Table 5, differences in respiration rates were observed between uncoated and coated apples. From the beginning of storage, control apples produced significantly more ethylene, approximately twice as much, as coated apples, suggesting that the coatings limited ethylene production and thus slowed the ripening process. It is known that the rapid increase in ethylene synthesis, which can occur in climacteric fruit, causes changes in colour, aroma, texture, and flavour. This increase can also reduce fruit firmness and, therefore, consumer acceptability [104]. A gradual increase in ethylene release was also observed for all coated samples up to 28 days of storage, whereas for uncoated samples, only up to 21 days, suggesting the limit of ripeness. The effectiveness of reducing ethylene production may depend on the concentration and type of phenolic acid contained in the coating. However, taking into account the coating type, all coated apples showed significantly lower respiration rates in comparison to control samples for each week of analysis ($p < 0.05$).

Table 5. Ethylene and carbon dioxide production of uncoated (Control) and coated apples with the coating solution based on apple (AP) incorporated with caffeic (AP_CFA) and protocatechuic (AP_PCA).

Time (Days)	Sample			
	Control	**AP**	**AP_CFA**	**AP_PCA**
	Ethylene (ppm C_2H_4/kg/h)			
0	$60.40 \pm 3.00^{b,A}$			
7	$63.80 \pm 7.10^{b,B}$	$18.25 \pm 0.65^{b,A}$	$8.85 \pm 1.15^{a,A}$	$6.80 \pm 0.10^{a,A}$
14	$67.00 \pm 1.70^{b,B}$	$15.50 \pm 0.55^{a,A}$	$13.70 \pm 2.30^{b,A}$	$11.85 \pm 1.00^{a,A}$
21	$67.65 \pm 7.45^{b,B}$	$28.75 \pm 1.05^{d,A}$	$25.25 \pm 3.45^{c,A}$	$23.25 \pm 1.85^{a,A}$
28	$52.75 \pm 3.05^{a,B}$	$25.70 \pm 0.70^{c,A}$	$23.90 \pm 0.50^{c,A}$	$23.55 \pm 1.65^{a,A}$
	Carbon dioxide (mg/kg/h)			
0	$0.23 \pm 0.13^{c,A}$			
7	$0.27 \pm 0.05^{d,B}$	$0.21 \pm 0.01^{d,A}$	$0.16 \pm 0.01^{c,A}$	$0.17 \pm 0.04^{c,A}$
14	$0.14 \pm 0.00^{a,A}$	$0.15 \pm 0.02^{b,A}$	$0.15 \pm 0.02^{b,A}$	$0.18 \pm 0.02^{d,A}$
21	$0.16 \pm 0.02^{b,A}$	$0.18 \pm 0.04^{c,A}$	$0.14 \pm 0.01^{a,A}$	$0.14 \pm 0.03^{b,A}$
28	$0.14 \pm 0.02^{a,A}$	$0.13 \pm 0.02^{a,A}$	$0.15 \pm 0.01^{b,A}$	$0.13 \pm 0.01^{a,A}$

Mean values ± standard deviations. Different superscript letters ($^{a–d}$) within the same column or rows (^{A,B}) indicate significant differences between the samples ($p < 0.05$).

Carbon dioxide is a compound released during fruit respiration. In the experiment, increased production of this gas was observed in the first week for the control sample, indicating vigorous respiration (Table 5). The use of coatings slowed down this process. The study also noted the impact of coatings on limiting the fruit's respiration process, as higher oxygen content in the chamber meant less absorption by the apple of this gas, which is used in the respiration process. This fact again confirms the improved barrier properties of coatings using phenolic acids. However, the coating type has no significant effect on carbon dioxide production compared to control samples at each time of analysis ($p < 0.05$), except at 7 days, when the highest value was observed for uncoated apples.

Yellow apples of the cultivars 'Tsugaru', 'Summer King', and 'Shinano Gold' (*Malus* × *domestica* Borkh.) were coated with a mixture of edible sucrose monoesters of fatty acids and ethanol and stored for up to 28 days at room temperature. A significant reduction in apple respiration rate was observed after both 14 and 28 days of storage, along with the preservation of skin colour and firmness [105]. Softening is also strongly correlated with increased respiration and ethylene production, as evidenced in a study of seven apple cultivars [98]. Coating 'Golden Reinders' and 'Granny Smith' apples with hydroxypropylmethylcellulose coating delayed fruit ripening by inhibiting ethylene production [79]. Ou et al. [106] demonstrated that the addition of ferulic acid to soy protein isolate reduced oxygen permeability by 18.8%. Similarly, Fabra et al. [107] observed a 32% decrease in oxygen permeability through sodium caseinate films after the addition of ferulic acid.

4.10. The Effect of Bioactive Coatings Based on Apple Pectin and Phenolic Acids on the Sensory Attributes of Apples During Storage

The sensory quality of apples is influenced by various factors, including hardness, texture, sweetness, tartness, aroma, and taste. A significant portion of the fruit's aroma is attributed to volatile compounds produced by the apples. For Golden Delicious apples, butyl acetate, hexyl acetate, 2-methylbutyl acetate, and ethyl 2-methylbutanoate are the most crucial compounds determining sensory quality [108]. Apple varieties with crisp flesh are assumed to have a higher concentration of water-soluble pectin, whereas less firm fruits contain more ion-bound pectin. Changes in cell wall hydrolase activity and related gene expression levels significantly affect fruit firmness. During fruit softening, polysaccharide modification depends on various enzymes, such as polygalacturonase, which is responsible for pectin decomposition [109]. In addition to regulating the sensory quality of fruits, the goal is to obtain edible coatings that are tasteless, odourless, and colourless [110]. Improved sensory characteristics, including texture, taste, aroma, and sweetness, were observed in apples treated with composite coatings made from arabinoxylan and β-glucan stearic acid ester, compared to uncoated apples. The fruits were stored at 22 °C for 45 days [111].

Figure 6 presents the sensory analysis results of control apples and apples coated with edible coatings containing phenolic acids after 7 and 28 days of storage. The results indicate that the sensory acceptability of all analysed variants decreased over time, as evidenced by lower scores after 28 days. After 7 days of storage, coated apples exhibited a better appearance than control apples. This was probably due to lower water loss, preservation of fruit firmness, and greater gloss resulting from the application of coating solutions, which enhanced the fruit's attractiveness. No major differences were noted in the smell between the samples, while the control apples received the best scores for taste. After 28 days of storage, apples coated with an edible coating containing protocatechuic acid received the highest scores, excluding the smell variant. However, the differences between this variant and the control were 0.5–1, suggesting no significant differences but only consumer preferences. Sensory data showed that coated apples were rated as juicier than uncoated apples. Coated apples were rated as crispy on the first bite and firm internally, whereas uncoated apples were rated as mushy and floury. Internal texture was defined by the

degree to which the apple disintegrated or remained intact. Control apples and those coated with coatings containing phenolic acids were harder than apples coated solely with apple pectin solution, which were the most crisp and floury. Therefore, it can be concluded that the coatings reduced the respiration rate of the apples, thereby preventing degradation. After 28 days of storage, apples coated with protocatechuic acid received the highest scores. Only their aroma was lower, possibly because the ripest control apples had a more intense aroma. The control apples exhibited a softer, drier texture and a matte appearance.

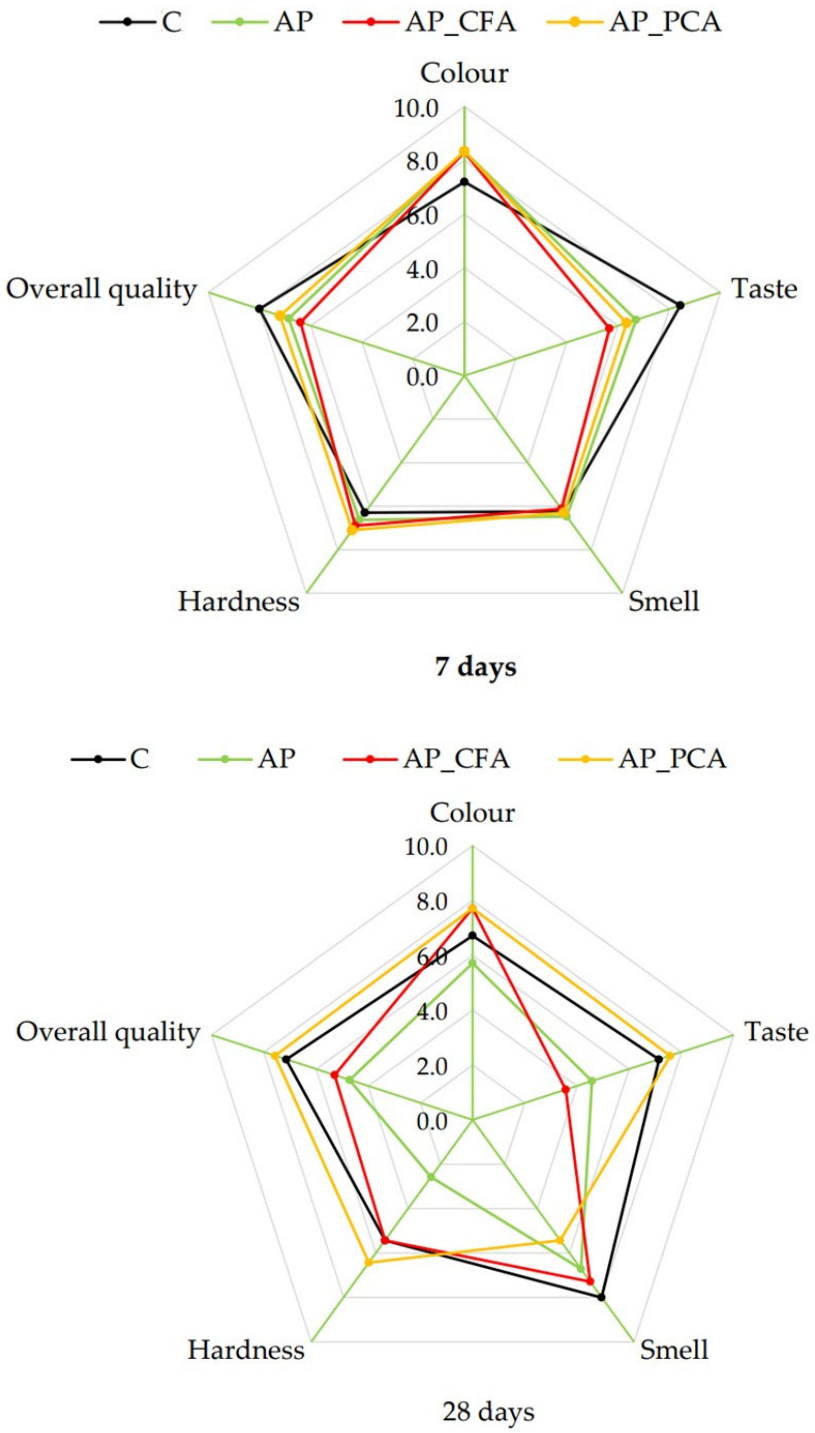


Figure 6. Sensory attributes of uncoated (C) and coated apples with the coating solution based on apple (AP) incorporated with caffeic (AP_CFA) and protocatechuic (AP_PCA) after 7 and 28 days of storage.

5. Conclusions

Bioactive coatings, based on apple pectin with and without the addition of selected phenolic acids, were developed and applied to Golden Delicious apples for preservation during storage at ambient conditions. Coated apples maintained appropriate fruit quality for 28 days of storage, without visible microbial mould growth. The coating process helped maintain fruit firmness and limited changes in apple mass during storage at 22 ± 1 °C and 40 ± 5% relative humidity. Apples treated with edible coatings exhibited a slower respiration process, resulting in delayed fruit ripening. This was confirmed by differences in total soluble solids, acidity, and pH of the apples. Coatings made of apple pectin alone, or apple pectin with added phenolic acids, preserved the apples' appropriate colour and visual appearance. All samples showed good stability over time. The coating reduced the rate of respiration and ethylene production, thereby delaying fruit ripening. Sensory analysis revealed that respondents consistently perceived coated apples in a positive light throughout the entire storage period. The highest taste scores were observed after 28 days of storage for the apple-treated solution containing protocatechuic acid. The differences in the results among the coated samples suggest that apple pectin coatings, even without the addition of active ingredients, were effective in maintaining the quality of apples. More research is needed to provide a more comprehensive understanding of the effects of edible coatings, including bioactive compounds in apples such as vitamin C and polyphenols, as well as radical scavenging activity and microbiological analyses. Further research is also required to investigate the combination of both phenolic acids to determine synergistic effects on antioxidant or antimicrobial activity, including release kinetics.

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