

Chemical and Sensory Quality Preservation in Coated Almonds with the Addition of Antioxidants

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Abstract: Almonds provide many benefits such as preventing heart disease due to their high content of oleic fatty acid-rich oil and other important nutrients. However, they are susceptible to oxidation reactions causing rancidity during storage. The objective of this work was to evaluate the chemical and sensory quality preservation of almonds coated with carboxymethyl cellulose and with the addition of natural and synthetic antioxidants during storage. Four samples were prepared: almonds without coating (C), almonds coated with carboxymethyl cellulose (CMC), almonds coated with CMC supplemented with peanut skins extract (E), and almonds coated with CMC and supplemented with butylhydroxytoluene (BHT). Proximate composition and fatty acid profile were determined on raw almonds. Almond samples (C, CMC, E and BHT) were stored at 40 °C for 126 d. Lipid oxidation indicators: peroxide value (PV), conjugated dienes (CD), volatile compounds (hexanal and nonanal), and sensory attributes were determined for the stored samples. Samples showed small but significant increases in PV, CD, hexanal and nonanal contents, and intensity ratings of negative sensory attributes (oxidized and cardboard). C had the highest tendency to deterioration during storage. At the end of storage (126 d), C had the highest PV (3.90 meqO₂/kg), and BHT had the lowest PV (2.00 meqO₂/kg). CMC and E samples had similar intermediate PV values (2.69 and 2.57 meqO₂/kg, respectively). CMC coating and the addition of natural (peanut skin extract) and synthetic (BHT) antioxidants provide protection to the roasted almond product.

Keywords: almonds, antioxidants, coating, peanut skins, sensory

Practical Application: Almond is an energetic and highly nutritious food and its global consumption is increasing. Almonds have a high oil content and high proportion of oleic acid as well as many other benefits for disease prevention. Oxidation is the main cause of almond product deterioration. The addition of edible coatings such as carboxymethyl cellulose and natural antioxidants in almond products can make a significant contribution to prolonging their shelf life, while developing healthy and natural foods.

Introduction

Almonds are an energetic and highly nutritious food. They contain minerals such as phosphorus, potassium, magnesium, calcium, iron, zinc, and vitamins A, E, B1, and B2. Moreover, they are appreciated for their flavor and nutritional value; they are also used in medicines and cosmetics (Moayedí and others 2011). The largest world almond producer is the United States (about 37%), followed by Spain (11%), Australia (7%), Iran (5%), Morocco (5%), and Italy (5%). Global consumption of almonds has increased due to demand from the European Union, Canada, Russia, China, and India (USDA 2013).

Almonds are very versatile; they can be used in both sweet and salty preparations. They are consumed raw/roasted or as various food applications: flour, milk, butter, ice cream, caramelized, and so on. Almonds have a high oil content and a high proportion

of oleic fatty acids and present many benefits for preventing heart disease (Boue and others 2009). However, they are susceptible to oxidation reactions, becoming rancid during storage. Oxidation reactions result in the development of unpleasant odor, flavor, and color, and in the loss of nutrients (fat soluble vitamins, essential fatty acids, carotenoids, amino acids, and so on) (McClements and Decker 2010). The quality and stability of almonds depends not only on their initial composition, but also on handling practices during growing and harvesting, and on processing methods, packaging and storage (Buranasompoba and others 2007).

Edible films or coatings have been developed as an alternative in the field of packaging and food preservation (Embuscado and Huber 2009; Riveros and others 2013). Edible coating features and functions depend on the type of biopolymers used (proteins, polysaccharides, lipids) and their chemical and structural composition. These functions affect the quality preservation of the food on which they are applied. They mainly serve as a barrier to the transference of substances between the food and the environment (Embuscado and Huber 2009). The ability to incorporate active ingredients such as antioxidants is another important property of an edible film. These additives are able to maintain and improve product quality by increasing the shelf life (Embuscado and Huber 2009; Rojas Graü and others 2009; Baldwin and others 2011).

The most common coatings used on almonds are chocolate, or sugar and cinnamon (candied almonds). Some edible films such as whey protein isolate and shellac have also proved to have consumer

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acceptance on almonds (Lee and others 2002; Baldwin and others 2011).

Carboxymethyl cellulose is an anionic linear polysaccharide derived from cellulose. It is an important industrial polymer with a wide range of applications in flocculation, detergents, textiles, papers, foods, and drugs. Recently, it has been used in edible films on different foods, improving their shelf life (Baldwin and others 2011; Tongdeesoontorn and others 2011; Riveros and others 2013, 2015).

Antioxidants are compounds that, at low concentration, prevent the formation of free radicals; capture and inhibit the initiation of oxidation process; interfere with the propagation of these reactions; or act to reverse the oxidation process (Sleiman Figueroa and others 2002).

Peanut skins are a residue of the blanching process. They are generally used to feed livestock, but also have an interesting potential as a source of natural edible antioxidants (Nepote and others 2004a,b, 2005; Larrauri and others 2013). Peanut skins contain a high proportion of phenolic antioxidant substances such as phenolic acids, stilbenes (resveratrol) and flavonoids, the main phenolic compounds being proanthocyanidins (Nepote and others 2004a; Sobolev and others 2006; Francisco and Resurreccion 2009; Ma and others 2014). While the nutritional and antioxidant properties of peanut polyphenols have been demonstrated, there are few studies evaluating their preservative capacity when they are applied in edible coatings to foods, especially those with a high fat content such as almonds.

The objective of this work was to evaluate the chemical and sensory quality preservation of almonds coated with carboxymethyl cellulose together with the natural and synthetic antioxidants during storage.

Material and Methods

Materials

Non Pareil almonds (*Prunus amygdalus* Batsch) were from San Martín, Mendoza, Argentina, crop 2014. Carboxymethyl cellulose (CMC) was provided by the company "Todo Droga"

(Cordoba, Argentina). Skins were from "Runner" peanuts, provided by Lorenzati, Ruetsch y Cia., Ticino, Córdoba, Argentina, crop 2014.

Chemical composition of almonds

Moisture, lipid, protein, and ash contents were determined on raw almonds by AOAC methods (2010). Total carbohydrates were calculated by difference.

Fatty acid composition was determined on almond oil. Fatty acid methyl esters were prepared from almond oil by transmethylation with a solution of 30 g/L sulfuric acid in methanol. The fatty acid methyl esters of total lipids were analyzed on a Perkin Elmer Clarus 600 gas-liquid chromatograph (Waltham, Mass., U.S.A.) equipped with a flame ionization detector (FID). A SACTM-5 capillary column (30 m × 0.25 mm i.d., 0.25- μ m film thickness; C#24156, Supelco, Bellefonte, Pa., U.S.A.) was used. Separation, identification, and quantification of the fatty acid methyl esters were performed according to Riveros and others 2010.

Extraction of phenolic compounds from peanut skins

Phenolic compounds were obtained from peanut skins by solid-liquid extraction with ethanol-water (70:30 v:v). The filtered extract was dried, resuspended in distilled water, and partitioned with n-hexane in a separation funnel. Then, water fraction was partitioned with ethyl acetate. The ethyl acetate fraction was separated and dried in rotatory evaporator. This fraction had 701.24 mg phenols/g dry extract, expressed as gallic acid equivalent measured by Folin-Ciocalteu method (Nepote and others 2004a).

Almond product elaboration and storage conditions

Initially, almonds were roasted in oven at 130 °C during 45 min. The following products were prepared: Almonds without coating (C), almonds coated with carboxymethyl cellulose (CMC), almonds coated with CMC and supplemented with peanut skins extract (E), and almonds coated with CMC and supplemented with butylhydroxytoluene (BHT).

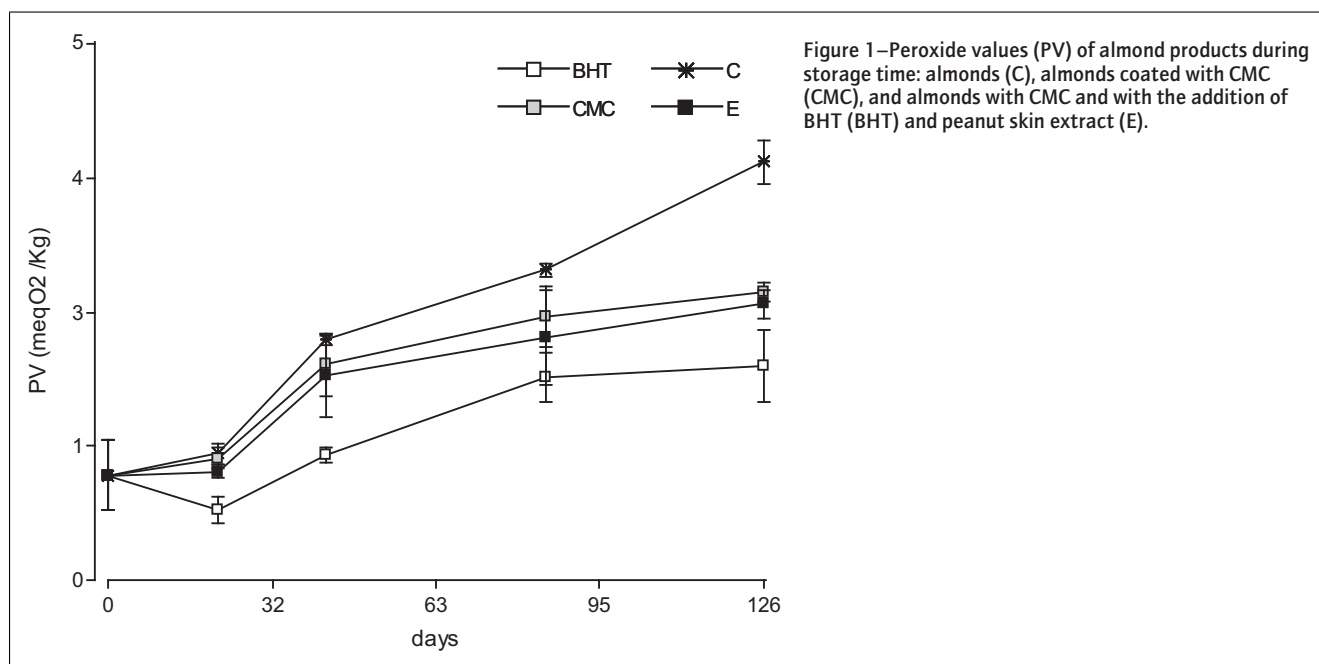


Figure 1—Peroxide values (PV) of almond products during storage time: almonds (C), almonds coated with CMC (CMC), and almonds with CMC and with the addition of BHT (BHT) and peanut skin extract (E).

CMC coating was prepared by mixing 0.5% carboxymethyl cellulose, 1.9% glycerol, and 97.6% distilled water. The solution was shaken and was used to cover almonds (4.5% final proportion in the product) (Riveros and others 2013).

Peanut skin extract and BHT were added into the coating solution before covering the almonds in 0.2% (Larrauri and others 2013) and 0.02% (CAA 2014), respectively, to obtain E and BHT samples.

After coating, all almond samples were dried in oven at 130 °C for 20 min.

Each almond product (C, CMC, E, and BHT) was stored in disposable polypropylene trays wrapped in PVC film, at 40 °C, during 126 d.

Lipid oxidation indicators, volatile compounds and sensory descriptive attributes were evaluated on almonds samples from storage at 0, 21, 42, 84, and 126 d.

Chemical analysis of almonds during storage

Almond oil was obtained by cold pressing from almond samples using a 20-ton press (HE-DU, Hermes I. Dupraz S.R.L., Córdoba, Argentina). The following indicators were determined on oil samples: peroxide value (PV) expressed as milliequivalents of active oxygen per kilogram of oil (meqO₂/kg) (AOAC 2010) and conjugated dienes (CD) expressed as extinction coefficient E (1%, 1 cm) (COI 2001).

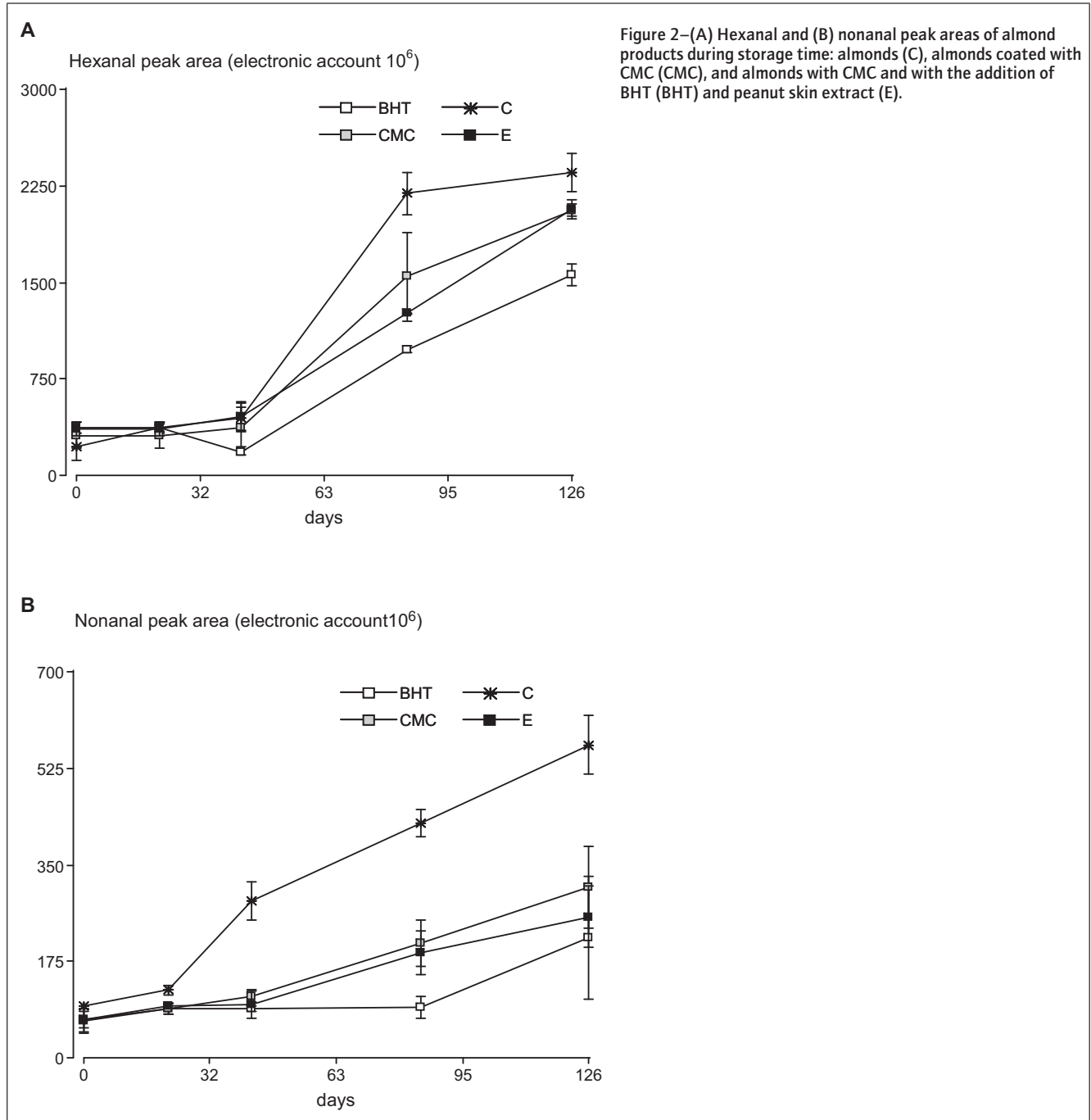


Figure 2—(A) Hexanal and (B) nonanal peak areas of almond products during storage time: almonds (C), almonds coated with CMC (CMC), and almonds with CMC and with the addition of BHT (BHT) and peanut skin extract (E).

Table 1—Intensity ratings (media ± standard deviation) of sensory attributes evaluated on fresh almond products (0 storage days): almonds (C), almonds coated with CMC (CMC), and almonds with CMC and with the addition of BHT (BHT), and peanut skin extract (E).

Sensory attributes	Almond products (intensity ratings 0–150)							
	C	*	CMC	*	BHT	*	E	*
Skin color	86.4 ± 2.7	a	89.4 ± 2.5	ab	89.4 ± 3.7	ab	91.2 ± 5.4	b
Internal color	42.8 ± 5.3		44.1 ± 5.8		44.9 ± 8.1		43.6 ± 5.7	
Glossiness	9.4 ± 3.1		8.5 ± 1.6		7.7 ± 2.1		8.9 ± 3.4	
Sweet	20.0 ± 3.1		20.6 ± 2.0		21.1 ± 1.9		19.4 ± 3.5	
Salty	5.0 ± 0.0		5.5 ± 1.6		5.3 ± 0.9		5.0 ± 0.0	
Sour	7.0 ± 4.5		7.0 ± 4.8		6.0 ± 2.1		7.0 ± 4.2	
Bitter	12.4 ± 2.5		11.6 ± 2.0		12.7 ± 3.4		11.9 ± 4.0	
Roasted	51.2 ± 8.5		53.7 ± 7.2		55.2 ± 7.9		52.2 ± 7.9	
Cardboard	0.0 ± 0.0		0.0 ± 0.0		0.0 ± 0.0		0.0 ± 0.0	
Oxidized	0.0 ± 0.0		0.0 ± 0.0		0.0 ± 0.0		0.0 ± 0.0	
Astringency	14.0 ± 4.2		13.2 ± 2.4		14.5 ± 3.2		13.4 ± 3.2	
Hardness	82.8 ± 9.1		83.2 ± 4.8		87.1 ± 5.8		85.3 ± 7.9	
Crunchiness	75.0 ± 7.1		75.4 ± 3.8		76.2 ± 2.0		74.9 ± 5.3	

*Different letters in the rows indicate significant differences between samples (ANOVA and LSD test, $\alpha = 0.05$).

Volatile analysis

Extraction of volatile compounds of almond samples was done by headspace solid phase microextraction (HS-SPME) and analyzed by gas chromatography/mass spectrometry (GC/MS) according to Quiroga and others 2015 in a Perkin Elmer Clarus 600 GC coupled with a mass detector (Perkin Elmer). The SPME fiber used was divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) 50/30 μm , StableFlex, 1-cm long (Supelco). ELITE 5MS (30 m \times 0.25 mm i.d., 0.25- μm film thickness; CN9316282, Perkin Elmer) column was used. Helium was the carrier gas; and ionization was performed by electron impact at 70 eV. Identification of volatile compounds in samples was performed in full scan mode (m/z 40 to 550) via a combination of the NIST mass spectral library and gas chromatographic retention times of standard compounds. When standards were not available, volatile compounds were tentatively identified using GC/MS spectra only. Chromatographic responses of detected volatile compounds (peak area electronic counts) were monitored for comparison of each compound between samples (Quiroga and others 2015).

Hexanal and nonanal were used as indicators for the characterization of off-flavors resulting from lipid peroxidation (Belitz and others 2009; Mexis and others 2009). Peak areas (electronic accounts) for hexanal and nonanal obtained by HS-SPME-GC/MS analysis of almond samples can be directly related to the content of these compounds in the samples. These peak areas were used to evaluate the changes in hexanal and nonanal contents in samples during storage (Quiroga and others 2015).

Sensory descriptive analysis of almonds during storage

A trained descriptive sensory panel (9 panelists: 7 women and 2 men) with at least 6 y of experience in evaluating peanut and almond products participated in the analysis. Panelists used a “hybrid” descriptive analysis method combining the quantitative descriptive analysis (Tragon Corp., Redwood City, Calif., U.S.A.) and Spectrum TM analysis (Sensory Spectrum, Inc., Chatham, N.J., U.S.A.) for evaluating samples using a 150 mm unstructured linear scale (Meilgaard and others 2006). All panelists were selected, trained, and calibrate according to Grosso and Resurreccion 2002. Thirteen attributes were evaluated on almond samples. Appearance attributes: skin color, internal color, and glossiness. Basic tastes: sweet, salty, sour, and bitter. Flavor attributes: roasted,

cardboard, and oxidized. Feeling factor: astringency. Texture attributes: hardness and crunchiness. A list of attribute definitions and a sheet with warm-up and reference intensity ratings were developed during the training sessions according to Riveros and others 2013.

All samples were evaluated in partitioned booths under fluorescent light at room temperature. Product samples (10 g) were placed in plastic cups with lids coded with 3-digit random numbers. A completely randomized block design was used for testing samples. The final lists of warm-up and reference intensity ratings and definitions were posted in the booths for all test sessions. The panelists were instructed to retest all references and the warm-up sample before evaluating the attribute intensity ratings of the roasted peanut samples. Data were registered on paper ballots (Riveros and others 2013).

Statistical analysis

The experiment was run in 3 repetitions. Data were analyzed using INFOSAT software Version 2013 (Facultad de Ciencias Agropecuarias, Univ. Nacional de Córdoba, Córdoba, Argentina). Means and standard deviations were calculated. Two-way analysis of variance (factors: “treatment” and “time”) and LSD Fisher’s multiple range test were developed to found significant differences among means in data from chemical and sensory analysis of almonds samples during storage ($\alpha = 0.05$). Pearson coefficients were estimated to establish correlations between dependent variables. Principal component analysis (PCA) was performed on the correlation matrix of standardized data from chemical and sensory variables. The purpose of the PCA was to explore associations between treatments, chemical and sensory variables.

Results and Discussion

Chemical composition of almonds

Raw almonds contained 55.25% \pm 0.20% lipids, 25.64% \pm 0.21% proteins, 16.81% carbohydrates, 2.62% \pm 0.07% moisture, and 2.30% \pm 0.09% ash.

The fatty acid composition of almond oil was: 71.25% \pm 0.36% oleic acid, 0.40% \pm 0.01% palmitoleic acid, 19.77% \pm 0.14% linoleic acid, 6.74% \pm 0.06% palmitic acid, and 1.84% \pm 0.58% stearic acid.

This composition is similar to that found in almonds from Argentina by other authors (Gayol and others 2009). The high

percentage of unsaturated acids means that almonds are susceptible to lipid oxidation deterioration.

Chemical analysis of almonds during storage

Peroxide values (PV) and conjugated diene values (CD) increased for all almond samples during storage. CD average values were between 0.00 (0 d) and 9.55 (126 d) for all samples, without significant differences between them. PV showed significant differences between samples during storage. The changes in peroxide values (PV) of almond products during storage are shown in Figure 1. Initially, all products had low PV (0.58 meqO₂/kg). Significant differences in PV between treatments were found from day 21. At the end of storage (126 d), the almonds without coating (C) had the highest PV (3.90 meqO₂/kg), and almonds with BHT had the lowest PV (2.00 meqO₂/kg). CMC (2.69 meqO₂/kg) and E (2.57 meqO₂/kg) samples had similar and intermediate PV. These results indicate that there is a protective effect of CMC, BHT, and E on almonds during storage. CMC edible film with BHT showed the highest protection effect. Similar behavior for these variables

were reported in previous studies in coated almonds (Gayol and others 2009) and high oleic peanuts (Nepote and others 2006a,b).

Riveros and others (2013) studied different edible coatings on roasted peanuts. They found that edible coatings preserve the sensory properties of the product, and carboxymethyl cellulose exhibited the best protective effect on roasted peanuts. Baldwin and Wood (2006) studied the use of edible coatings to preserve pecans, reporting that CMC-based coatings exhibited the potential to extend the shelf life of pecan kernels.

Previous studies on edible films enriched with antioxidant compounds reported protective effects on the food product studied (Eça and others 2014; Riveros and others 2015). Ponce and others 2008 studied different edible films (chitosan, CMC, among others) enriched with oleoresins. They reported that chitosan edible film enriched with rosemary and olive oleoresins improved the antioxidant protection of minimally processed squash, preventing browning reactions.

In other work, peanut skin extracts added to different products (sunflower oil, honey roasted peanuts and salami) exhibited

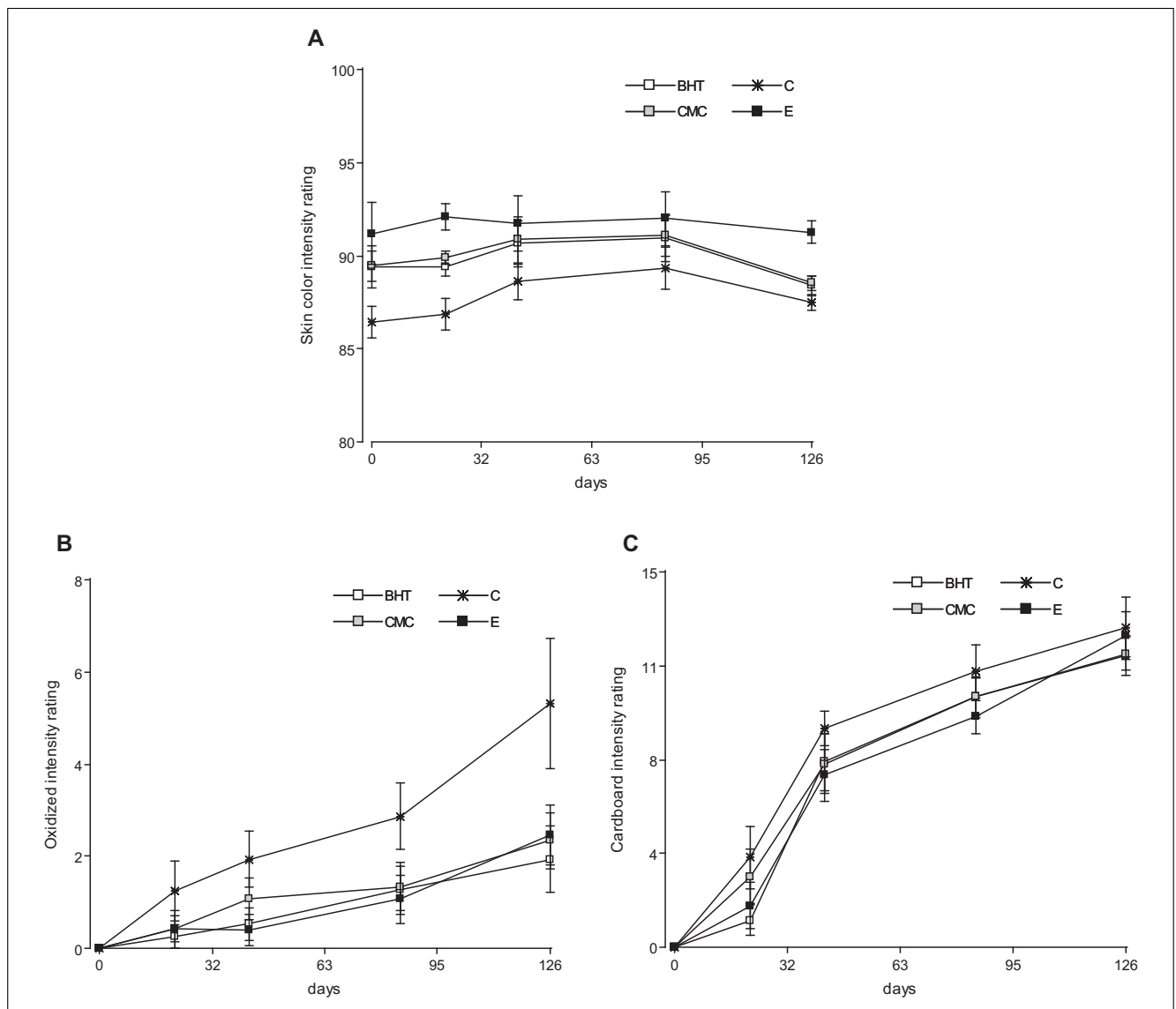


Figure 3—Intensity ratings of sensory attributes of almond products during storage time: almonds (C), almonds coated with CMC (CMC), and almonds with CMC and with the addition of BHT (BHT) and peanut skin extract (E). (A) Skin color, (B) oxidized, and (C) cardboard flavors.

antioxidant properties (Nepote and others 2002, 2004b; Larrauri and others 2013).

Volatile analysis

Different volatile compounds were detected in almond products by GC/MS analysis. Hexanal and nonanal were the main components that increased during storage (Figure 2). Those aldehydes are indicators of oxidation reactions. Hexanal content is directly related to the development of rancid flavors in lipid-rich foods (Mexis and others 2009; Quiroga and others 2015). Initially, all almond treatments had similar hexanal and nonanal contents (peak areas from 219×10^6 to 373×10^6 for hexanal and from 67×10^6 to 94×10^6 for nonanal). Significant differences in the peak areas between almond treatments were found from day 84 (Figure 2). C samples presented higher hexanal and nonanal peak areas. On the other hand, BHT and E showed lower values for these volatiles.

Lee and others (2014) studied the influence of storage on the volatile profiles on roasted almonds. They also reported increased hexanal in samples during storage at 20 and 35 °C. In addition, Xiao and others (2014) showed increased hexanal, nonanal and other aldehydes associated with increased roasting time in almonds.

Sensory descriptive analysis of almonds during storage

The different sensory attributes evaluated for fresh almond products (0 storage days) are shown in Table 1. In general, almond products were characterized by high intensity of skin color (86.4 to 91.2), hardness (82.8 to 87.1), and crunchiness (74.9 to 76.2).

Products had roasted flavor intensity ratings between 51.2 and 55.2. Negative attributes such as oxidized and cardboard flavors were not detected in fresh products. Skin color was the only attribute that showed significant differences between samples. C (86.4) had lower skin color intensity rating than E (91.2). Almonds with coating (CMC, BHT, and E) showed similar color intensities. The CMC coating makes the product darker.

Changes in sensory attributes of almond products during storage are shown in Figure 3. In general, almond products showed few changes in their sensory attributes. Samples had initial differences in skin color, but these differences remained constant during storage time. Oxidized and cardboard flavors were the only attributes with significant increases during storage. Oxidized flavor was rated as being different between samples from storage day 84. C exhibited higher intensity rating than the other treatments. CMC, E, and BHT did not have significant differences in their oxidized flavor during storage. Cardboard flavor increased during storage in all samples, without significant differences between them.

In previous work on peanut products (Nepote and others 2004a,b, 2006a,b), intensity ratings of oxidized and cardboard flavors increased during storage. Those flavors were related to volatile oxidation compounds (hydroperoxides, aldehydes, ketones, and so on).

Principal component analysis

A biplot from the first and second principal components of PCA is shown in Figure 4, considering the variables PV, CD,

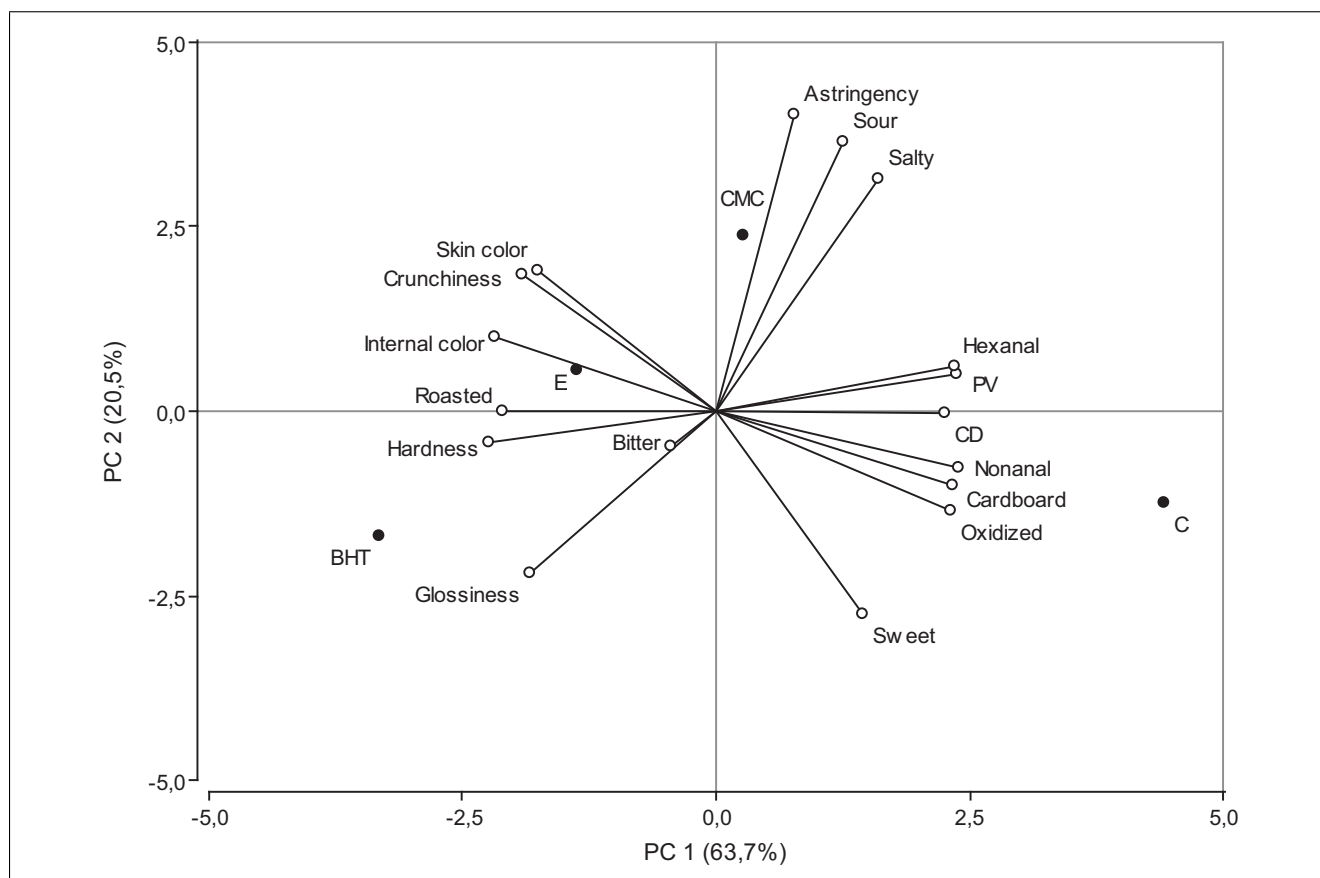


Figure 4—Biplot from first (PC 1) and second (PC 2) principal components of PCA. Variables: peroxide value (PV), conjugated dienes (CD), hexanal, nonanal; and sensory variables evaluated on different almond products during storage: almonds (C), almonds coated with CMC (CMC), and almonds with CMC and with the addition of BHT (BHT) and peanut skin extract (E).

Table 2—Significant Pearson's correlation coefficients ($P < 0.01$) for chemical and sensory variables on almond samples from storage.

	PV	CD	Hexanal	Nonanal
CD	0.67			
Hexanal	0.85	0.81		
Nonanal	0.76	0.71	0.82	
Cardboard	0.76	0.74	0.88	0.69

hexanal, nonanal, and sensory attributes from almond treatments during storage. Those components explained 84.2% of the total variability. The variables: PV, CD, hexanal, nonanal, oxidized, and cardboard are found on the right side of the biplot which indicates positive correlation between them. On this side of the plot, C was associated with higher values of those variables, mainly with nonanal, cardboard, and oxidized. CMC was mainly associated with the sensory attributes: astringency, sour, and salty. The sensory variables: roasted, hardness, skin color, internal color, crunchiness, glossiness, bitterness, and the BHT and E treatments were located on the left hand side. The BHT sample was mainly associated with hardness and glossiness while the E treatment was found near internal color, roasted, skin color, and crunchiness.

According to correlation analysis (using Pearson coefficients), positive correlations were found between CD, PV, hexanal, nonanal, and cardboard (Table 2).

In previous studies, relationships between chemical and sensory variables were reported for peanut products (Nepote and others 2006a,b; Olmedo and others 2009). Grosso and Resurreccion (2002) reported a correlation between oxidized and painty flavors, and hexanal content on cracker-coated and roasted peanuts. They used regression equations to define relationships between the end point of consumer acceptance and flavor acceptance from sensory descriptive analysis and hexanal measurements. In other research, hexanal and other aldehydes were also associated with oxidation reactions (Mexis and others 2009; Quiroga and others 2015). Quiroga and others (2015) reported a positive association between hexanal content, oxidation indicators (peroxide and p-anisidine values) and negative attributes (oxidized and cardboard) of roasted sunflower seeds.

Conclusions

The results in this study indicate that roasted almonds show an increase in lipid oxidation indicators (peroxide value and conjugated dienes) and in the intensity ratings of negative sensory attributes like oxidized and cardboard on storage. Carboxymethyl cellulose coating and the addition of natural (peanut skin extract) and synthetic (BHT) antioxidants provide protection against this oxidative deterioration in roasted almonds. BHT exhibits the highest antioxidant activity for this product. The addition of an edible coating and natural antioxidant can make a significant contribution to extending the shelf life of different food products, meeting the needs of demanding consumers looking for healthy and natural foods.

Acknowledgments

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