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Novel active biopackaging incorporated with macerate of carob (*Ceratonia siliqua* L.) to extend shelf-life of stored Atlantic salmon fillets (*Salmo salar* L.).

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ABSTRACT

Two antioxidant bio-based packaging materials incorporated with an 8% aqueous solution of carob seed ethanol macerate (CSE) or 8% aqueous solution of carob seed acetone macerate (CSA) were developed. Fresh salmon fillets were packaged in active and control films and stored at refrigeration temperature at 4 ± 1 °C. The quality was evaluated by sensory analysis, color, pH, water holding capacity and drip loss, thiobarbituric acid reactive substances, and total volatile basic nitrogen. The CSE and CSA samples presented satisfactory off-odor and overall acceptability results than the control samples until the 5th storage day. For color analysis, active samples preserved better the characteristics of fresh salmon than the controls during the first storage days. The fresh salmon fillet covered with active packaging presented on the 5th storage day a lower values of pH (6.54 ± 0.05 and 6.60 ± 0.11), drip loss (3.17 ± 0.76 and 2.83 ± 0.29), thiobarbituric acid reactive substances (0.056 ± 0.033 and 0.088 ± 0.054) and total volatile basic nitrogen (30.04 ± 3.54 and 32.67 ± 4.81), whereas, the highest water holding capacity values (92.23 ± 1.09 and 92.91 ± 3.07) for CSE and CSA respectively, as compared to those of blank biopackaging.

1. Introduction

Fresh Atlantic salmon fillets (*Salmo salar* L.) are popular seafood consumed worldwide because of their household convenience, high nutritional value, and beneficial health effects primarily due to the presence of omega-3 long-chain fatty acids, such as eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), which are essential and vital nutrients (Bell, Henderson, Tocher, & Sargent, 2004). However, due to their polyunsaturated fatty acids, fresh salmon fillets are easily susceptible to lipid oxidation, even at refrigeration temperatures. The oxidation process decreases the salmon quality and modifies the taste, odor, texture, and consistency. At the same time, its nutritional value decreases (Ghaly, Dave, Budge, & Brooks, 2010). Over the last decades, different searches for technologies that favor fresh salmon utilization have been intensified due to the increasing demand for high-quality fresh salmon (Fernández & Roeckel, 2009).

The improvement in preservation techniques to bring the fresh salmon safely to the consumer, simultaneously maintaining its organoleptic characteristics, are the main allies of fish industries (Soares, Silva, Barbosa, Pinheiro, & Vicente, 2017). Various works have been published applying natural antioxidants to fish preservation (Abidar et al., 2020; Djenane, 2015). Nevertheless, the direct addition of natural antioxidants into food formulations is challenging because they tend to be less potent than synthetic additives and therefore must be added in more significant amounts, which may negatively affect the organoleptic properties of the product (Nerín, Aznar, & Carrizo, 2016). Natural antioxidants could be added indirectly into the biodegradable polymer matrix. This novel strategy is known as active biopackaging (Djebari et al., 2021; Wrona, Vera, Pezo, & Nerín, 2017). It appears to be a pivotal blueprint for reducing the usage of synthetic antioxidants and consequently minimizing the astringency and bitterness of these compounds. Moreover, the application of biopackaging reduces the environmental impact of conventional packaging (Khwaldia, Ferez, Banon, Desobry, & Hardy, 2004).

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The food processing industry generates large amounts of waste, which are considered a source of natural bioactive compounds (Zhao, Xiong, & McNear, 2013). In this context, efforts are focused on their valorization as a source of natural antioxidants (Lammi et al., 2017; Lammi, Le Moigne, Djenane, Gontard, & Angellier-Coussy, 2018).

Native to the Middle East, the carob tree (*Ceratonia siliqua* L.) is found naturally in Algeria, Spain, and other Mediterranean countries (Quezel & Santa, 1963). Carob seeds makeup 10% of total fruit weight and are considered an essential by-product that can be used in active biopackaging materials for the food industry applications. Carob seeds are a source of tocopherols and organic acids, including phenolic compounds. Antioxidant properties attributed to their bioactive compounds have been shown (Ben Ayache et al., 2020; Fidan et al., 2020).

Various *in vivo* studies have shown the antioxidant effectiveness of active antioxidant biopackaging based on biodegradable polymer materials on the shelf-life of Atlantic salmon (Cao & Song, 2020; Lan et al., 2021). Goulas et al. (2019) showed that the application of carob polyphenolic coating on the salmon produced an antioxidant effect by reducing the oxidation phenomena. It has been previously reported that when the primary free radicals are eliminated, the release of antioxidants is not required. Thus, owing to its strong radical scavenging activity, carob seed extract can be an efficient antioxidant applied without direct contact with the food matrix. Furthermore, Ait Ouahioune et al. (2022) demonstrated the strong antioxidant activities of new antioxidant biopackaging based on cellulose material incorporated with carob seeds macerates. Moreover, a migration study of antioxidants from the materials was performed according to the European Regulation EU/10/2011 for food contact materials. The obtained results showed no migrants, either in the case of the volatile or non-volatile compounds. Also, high antioxidant capacity as a free radical scavenger based on the study of Pezo, Salafranca, and Nerín (2006); Pezo, Salafranca, and Nerín (2008) has been demonstrated.

To the best of our knowledge, macerated carob seeds have not been tested as a potentially active agent in antioxidant biopackaging to extend the shelf-life of stored fresh Atlantic salmon (FAS).

The purpose of this study was to determine the antioxidant efficiency of the new antioxidant active biopackaging material containing carob seeds macerates for the stored FAS, and therefore extend the shelf-life of this product during long term storage at 4 ± 1 °C. Organoleptic assay, color, drip loss, and water holding capacity (WHC), pH, thiobarbituric acid reactive substances (TBARS), and the total volatile basic nitrogen (TVB-N) will be evaluated.

2. Material and methods

2.1. Chemicals

Trichloroacetic acid (99%, CAS 76-03-9) was provided by Sigma Aldrich (Madrid, Spain); malondialdehyde-tetrabutylammonium salt (98%, CAS 100683-54-3) and 2-thiobarbituric acid (TBA ≥98%, CAS 504-17-6) were purchased from Fluka (Madrid, Spain). Ethanol (high-performance liquid chromatography (HPLC grade, CAS 64-17-5) and acetone (UV, IR, HPLC, GPC, APS, CAS 67-64-1)) were from PanReac, AppliChem (Germany). Sodium hydroxide (NaOH, 0.25 N, CAS 1310-73-2) and sulfuric acid (H_2SO_4, 96%, CAS 7664-93-9) were from PanReac Quimica SLU (Barcelona, Spain). Methyl blue (CAS 28983-56-4), methyl red (CAS: 493-52-7), and potassium carbonate ($K_2CO_3 \geq 99\%$, CAS: 584-08-7) were obtained from Sigma-Aldrich Química S.A. (Madrid, Spain). Ultrapure water was obtained from a Wasserlab Ultramatic GR system (Barbatáin, Spain).

2.2. Antioxidant agents

Samples of carob fruit (*Ceratonia siliqua* L.) were collected during December 2018 from a carob tree located in Tizi-Ouzou (Algeria, Coordinates: 36°43′N 4°3′E), and an amount of 60 g of carob seeds (CS) was manually separated from the fruits and air-dried at room temperature (≈27 °C) for one month. Then, dried seeds were ground with an electric grinder. As extracting solvent, 80% aqueous solution of ethanol or 80% aqueous solution of acetone by maceration method from the seeds at room temperature were used according to Adilah, Jamilah, Noranizan, and Hanani (2018) with slight modifications. This process was successively repeated three times with the renewal of the solvent each 24 h. The macerates were filtered using Whatman filter paper (porosity 0.22 μm) and were stored in glass bottles in the dark at 4 ± 1 °C until further use. The following abbreviations of macerates were applied: CSE means seeds macerated with 80% ethanol; CSA means seeds macerated with 80% acetone.

2.3. Sample preparation fillet

Fresh salmon fillet (FSF) was selected to be used in the antioxidant effectiveness assays. The product was purchased whole within 24 h post-harvesting from a local seafood market in Zaragoza, Spain. Salmon was beheaded, and the bones were removed from it immediately. After that, it was cut into 23 identical fillets that weighed approximately 1500 g. The salmon slices were then transported to the analytical chemistry laboratory (Zaragoza University, Campus Rio Ebro, Spain) in a polystyrene closed box with appropriate flake ice within 10 min of arrival. The salmon slices were divided into four batches (each batch for one treatment). The skin was removed from the flesh in aseptic conditions. Slices of 22 g were prepared in triplicate for each treatment during the different storage days. A sterile knife was used for samples preparation. All the prepared samples were kept on ice until utilization to avoid deterioration. All the experiments were carried out using the same initial fresh salmon to ensure the same product quality.

2.4. Biopackaging active material

The developed packaging material was based on two cellulose (CL) polymer layers from the Nutraflex (45NK) product range supplied by Futamura UK Ltd (Burgos, Spain) laminated together with a water-based biodegradable adhesive for food packaging applications from Samtack (Barcelona, Spain). Solution (w/w) of CS macerates at a concentration of 8% in water-based biodegradable adhesive were prepared and vortexed for 1 min until complete homogenization. The active adhesive was spread on the CL sheet using the coating machine K control coater from RK print coat instruments (Litlington, UK). Wire close wound bar (Bar number: 4; color code: black; wire diameter: 0.51 mm; wet film deposit: 40 μM) was used for coating. The CL sheet was air-dried to get rid of the solvent. The CL sheet with dry adhesive was covered by another CL layer. The developed multilayer biomaterial was placed in BiO 330 A3 Heavy Duty Laminator (South Korea), and it was pressed at 40 °C with velocity number 5.

Solution (w/w) of ethanol (C1) and acetone (C2) were prepared at a concentration of 8% in water-based biodegradable adhesive as the blank biomaterial.

An amount of 22 g of FSF was placed in the Petri dish (Ø = 10 cm) covered with a 10 cm × 10 cm sheet of each active biopackaging. The sample size (n-value) for each treatment was fifteen (n = 15). The experiments were carried out without direct contact between the food sample and the active agent to simulate the most real conditions for fish packaging. Each petri dish was then carefully introduced in a cellulose bag. Also, the salmon with blank biomaterial without an active agent (C1 and C2) was prepared to compare the effect of fish spoilage. The samples were hermetically thermo-sealed and kept at 4 ± 1 °C for 13 days. All samples were prepared in triplicate and analyzed after 0, 3, 5, 8, and 13 days.

2.5. Quality assessment

The quality of the FSF was tested during the experiments by evaluating different organoleptic properties and physicochemical parameters: pH, drip loss and WHC, color measurement, TBARS test, and TVB-N values. All the measurements were done in triplicate and performed after 0, 3, 5, 8, and 13 days of samples storage.

2.5.1. Sensory analysis

The sensory properties that were considered to discriminate between the samples for a grade of acceptability were the visual appearance of the salmon, such as color and texture, and the odor attribute, which are directly related to the consumers salmon acceptability, according to the method described in a previous study (Wrona, Vera, et al., 2017). Fresh salmon should have a bright pink or orange color. If salmon fish has a pale, dull color, means that it is likely spoiled. Salmon fish should also have fine white lines running through it, which indicate freshness.

The sensory analysis of FSF was done by five-member trained panelists selected among the workers of the analytical chemistry laboratory (University of Zaragoza, Spain). They were trained according to the method described by (Shahidi & Botta, 1994). The evaluation was based on two sensory attributes, namely off-odor and overall acceptability of FSF. The analysis consisted of 24 evaluations of salmon samples coded with random numbers. The off-odor and overall acceptability were evaluated using a 5-point scale according to Djenane, Sánchez-Escalante, Beltrán, and Roncalés (2001). Scores for off-odor referred to the intensity of odors associated to fish oxidation were as follows: 1 = none; 2 = slight; 3 = small; 4 = moderate; and 5 = extreme and were evaluated immediately after opening the package with sample. While evaluating the acceptability, 5-point hedonic scale was used, where 1 = dislike extremely; 2 = dislike; 3 = nor like or dislike; 4 = like; 5 = like extremely. Results are expressed as the predominant score given by panelists.

2.5.2. Color measurement

A colorimeter Chroma Meter CR-400 from Konica Minolta (Tokyo, Japan) with D65 as the light source was used to measure the color on the surface of salmon fillets. Each packaging was opened, samples were removed, and left for blooming for 15 min. Eighteen color determinations for each replicate were performed to cover the whole surface. CIE L* (lightness), a* (redness), and b* (yellowness) were used for the characterization of the color. The equipment was calibrated daily with white chroma meter standard plate (Y = 93.7; x = 0.3130; y = 0.3191). For each sample, the total color difference (ΔE) as an estimate of color changes was determined following equation (1):

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

The color values of fresh salmon on 0 storage day were used as reference values for ΔE calculation (L_0^*, a_0^*, b_0^*).

2.5.3. pH measurement

The pH of the salmon samples was determined according to the method described by Sallam (2007). A 5.0 g of fish sample were cut into small pieces and then homogenized with 10 mL of distilled water in a cup-blender for 30 s. Then, the pH of the resulting homogenate was measured at room temperature 25 °C using a digital pH-meter GLP 22 from Crison Instruments (Barcelona, Spain) previously calibrated at pH 4 and 7.

2.5.4. Drip loss and water holding capacity

Drip loss in salmon samples for the different storage days was expressed as the difference in fillet weight between day 0 (m_0) and day x (m_x) and was calculated according to equation (2)

$$DL = (m_0 - m_x/m_0) \times 100 \quad (2)$$

WHC was measured in the dorsal muscle from all salmon fillets at 0, 3, 5, 8, and 13 storage days. The dry content (D_0) of the muscle was determined by drying 2 g of each sample for 24 h at 105 °C until the equilibrium weight was obtained; hence water content (V_0) was determined (ISO-6496, 1999). Meanwhile, a piece of salmon was weighed (2 g) and placed in a tube with a pre-weighed filter paper (Whatman N °1). The tubes were then centrifuged using CENTROMIX model S-549 from JP Selecta (Barcelona, Spain) at rpm 30*100 for 15 min at room temperature. The exudate filtered through the filter paper was collected at the bottom of the centrifuge tube. The samples were weighed before and after this procedure. The results were expressed as the amount of sample remaining after centrifugation and were calculated according to equation (3):

$$WHC = (W_0 - \Delta W / W_0) \times 100 \quad (3)$$

where

$$W_0 = V_0/(V_0 + D_0) \times 100 \quad (4)$$

$$\Delta W = \Delta V_0/(V_0 + D_0) \times 100 \quad (5)$$

V_0 = the initial water content of the muscle.

D_0 = the initial dry matter of the muscle.

ΔV_0 = the weight of the liquid separated from the sample during centrifugation

2.5.5. Oxidative stability

The lipid oxidation study of salmon samples was performed by the TBARS method as described by Djenane, Aboudaou, Ferhat, Ouelhadj, and Ariño (2019). Briefly, 10 g of FSF were mixed with 40 mL of a 10% aqueous solution of trichloroacetic acid (TCA) until a homogeneous suspension was obtained. The supernatant was filtered using Whatman N^{o}1 filter paper. Then, 2 mL of the filtrate were mixed with 2 mL of an aqueous solution of thiobarbituric acid (TBA) at a concentration of 20 mM. The mixture was heated to 97 °C for 20 min and then cooled to room temperature. The absorbance was measured at 532 nm using a spectrophotometer UV-1700 (Shimadzu Pharmaspec Iberica, Madrid, Spain) against a reference blank containing the TBA reagent. All the measurements were prepared in triplicate. To calculate the concentration of secondary lipid oxidation product, a calibration curve was prepared using a malondialdehyde solution (MDA) in the range 0.1 − 0.8 μg/g. Results were expressed as mg of MDA per kg of fish.

2.5.6. TVB-N content

The TVB-N content was determined using the Conway micro diffusion method. A slice of salmon (4 g) was weighed, transferred to the stomacher bag, and homogenized with 15 mL of water for 2 min at 265 rpm. Then, 10 mL of 10% TCA (w/v) were added and homogenized for 4 min to eliminate the protein content. The slurry was collected, filtered, and centrifuged. The test was performed using the micro-diffusion chamber of Conway. Briefly, 1 mL of a saturated potassium carbonate solution was placed in the outermost area of the chamber and mixed with 1 mL of the supernatant. One mL of sulfuric acid was added to the central compartment of the chamber. Finally, the chamber was closed, sealed, and carefully mixed by circular movement, avoiding mixing the liquids in different compartments. After that, incubation was performed at 35 °C for 1 h. The sulfuric acid was titrated by a 0.1 N solution of sodium hydroxide using a micro-burette. Two drops of indicator solution were added (methyl red-methylene blue indicator).

The amount of TVB-N was calculated by equation (6).

$$TVB\text{-}N = [(Vac - Vba) \times 0.14 \times 25/ (Vm \times PM)] \times 100 \quad (6)$$

where Vac is the volume of sulfuric acid (1 mL), Vba is the volume of NaOH consumed in the titration, Vm is the volume of sample added to the Conway cell (1 mL), and PM represents the salmon weight (4 g). The TVB-N was expressed as mg N/100 g fish.

2.6. Statistics

Experiments were performed at least in triplicate. The results were expressed as mean ± standard deviations. The statistical significance of differences among different treatment and storage periods was evaluated by one-way analysis of variance (ANOVA) followed by the Post Hoc HSD Tukey test with significance at $p < 0.05$. The sample size was $n = 15$. A correlation matrix (CM) of data was performed, which shows the quantitative assessment classes of severability. The normality and homogeneity of variance assumptions were tested and checked using Cochran's C test, Harley and Bartlett, with $P < 0.0063$.

Principal component analysis (PCA) was performed to reduce the multivariate data's dimensionality, visualize them graphically, with minimal loss of information, and identify different groups of FAS samples. This multivariate analysis allowed to summarize the information included in the variables studied into a few principal components or factors, providing a simplified interpretation of data variance through mathematical methods. All data were statistically evaluated using STATISTICA version 7.1 (Statsoft, Tulsa, OK, USA).

3. Results and discussion

3.1. Sensory analysis

In the present study, the cellulose biopolymer selected as the substrate is from natural resources and emerges as a response to growing attention to environmental pollution and environmental footprint. It was combined with active agents from carob seeds macerates that have antioxidant effects for the development of active bio-based packaging systems to improve the shelf-life of FAS. These compounds were considered agro-food waste (Santonocito et al., 2020). This approach is very interesting, as it creates an added value to this industrial by-product and reduces the total price of the final packaging system (Quiles-Carrillo, Mellinas, Garrigos, Balart, & Torres-Giner, 2019).

The results of the evaluation of the sensory characteristics such as smell and overall acceptability in the salmon fillet influenced by the developed multilayer active biopackaging on 0, 3, 5, 8, and 13 storage days at 4 ± 1 °C are shown in Fig. 1.

On the 3rd and 5th storage days, samples wrapped with both antioxidant biopackaging material were acceptable and had a pleasant odor, characteristic of fresh fish compared to samples covered with blank biopackaging material, which were directly rejected on the 5th storage day. Regarding the results after the 5th storage day, it can be observed that in all cases, samples packaged with CSE and CSA biomaterial presented lower scores of off-odor attributes and higher scores of the acceptability attributes than the corresponding salmon fillet packaged with blank biopackaging material. However, the scores of off-odor attributes increased significantly. In contrast, overall acceptability scores decreased significantly ($p < 0.05$) as the storage time increased. All samples presented off-odor and not acceptable characteristics and were considered spoiled, where the rancidity and ammonia fishy smell were the highest in the case of salmon covered with blank biomaterial. That might be caused by lipid and protein oxidation (Guyon, Meynier, & de Lamballerie, 2016). The scores recorded for smell and overall acceptability displayed that the addition of carob seeds macerates considerably protected the sensory characteristics of the salmon throughout the storage period.

This fact may be due to the presence of antioxidant compounds, particularly volatile compounds and non-volatile ones. Ait Ouahioune et al. (2022) identified the volatile compounds composition of carob seeds macerates and the results revealed that they were rich in volatile bioactive compounds characterized by antioxidant properties such as monoterpenes and sesquiterpenes, which have been proven to have an antioxidant capacity as free radical quenchers and act through either the hydrogen donor or electron donor mechanism (Djebari et al., 2021).

The odor effect of carob agent incorporated in the active biomaterial on samples packaged with CSE and CSA biomaterial were evaluated by comparing them to the blank samples, and no odor effect of carob seeds was detected in the samples of both kinds of active packaging, especially in the 3rd and 5th storage day.

3.2. Color analysis

The color of the salmon fillet surface is an important quality attribute for consumers' acceptability (Merlo et al., 2019). The data of the evaluation of color *CIE* $L^*a^*b^*$ throughout the storage time showed significant interaction ($p < 0.05$) between treatments and storage time (Table 1). The initial color parameters values obtained in the FSF were as follows: 49.55 ± 1.73 for L^* (lightness), 12.43 ± 1.52 for a^* (redness), and 15.41 ± 0.99 for b^* (yellowness). According to the literature, there are several reasons for color differences of salmon muscle, which different contents could cause in astaxanthin carotenoid and the haem pigments (Yagiz et al., 2010).

Fig. 2 shows the photos of the salmon samples on the 5th storage day. The three replicates of each sample look similar, and satisfactory reproducibility was obtained for them. Spoilage color resulting in the change of salmon color can be observed in the case of samples from blank packaging (C1 and C2), whereas the samples from active packaging (CSE and CSA) retained the reddish color of fresh salmon. After the 5th storage day, all samples exhibited a slight apparent increase in the lightness and yellowness parameters, whereas the redness parameter decreased during the storage period.

In contrast, as the storage time increased, the samples covered with CSE and CSA active packaging retained the color characteristics of fresh salmon (lower L^* and b^*-value and higher a^*-value) than the samples from blank packaging. This can be explained by the structural changes caused by protein denaturation, which increases the light absorption and scattering on the surface of the salmon fillet (Lerfall, Bendiksen, Olsen, & Østerlie, 2016; Merlo et al., 2019; Van Haute, Raes, Devlieghere, & Sampers, 2017).

Regarding the change in a^*-value parameter, this can be mainly attributed to the potential oxidation of the salmon pigments, responsible for the typical red-orange color of Atlantic salmon caused by carotenoids (mainly astaxanthin and canthaxanthin) besides haem proteins (Giménez, Roncalés, & Beltrán, 2005).

According to Ruff, FitzGerald, Cross, and Kerry (2002), the color muscle becomes more yellow as the malondialdehyde concentration resulting from lipid oxidation increases. In parallel, the yellowish color is related to the interaction between the aldehydes as products from a lipid auto-oxidation with amino groups of proteins. Similar phenomenon was obtained in previous studies (Giménez et al., 2005; Merlo et al., 2019). This is reflected in the value of the calculated color change (ΔE) parameter between fresh salmon and samples from different storage days. As described by Tiwari, Muthukumarappan, O'Donnell, and Cullen (2008), the total color difference can be perceived as small for $\Delta E < 1.5$, distinct for ΔE between 1.5 and 3, and very distinct for $\Delta E > 3$. Thus, the changes in salmon color could be classified as small for the active samples on the 5th storage day, whereas very distinct and visible for the human eyes on the last storage day, being similar to control samples (Table 1). Therefore, the developed active bio-based packaging incorporated with macerates of carob seeds as agro-food waste had a certain preservative effect on the color by minimizing the oxidation of pigment of salmon, which may be due to the presence of active compounds present in CSE and CSA that eliminate the free radicals responsible for the initiation of the oxidation phenomenon from the package headspace, thus avoiding the direct contact of active agents with the packaged FSF (Carrizo et al., 2015).

3.3. pH

The pH measurement is used as a spoilage indicator in fishery products and can provide interesting information on the state of

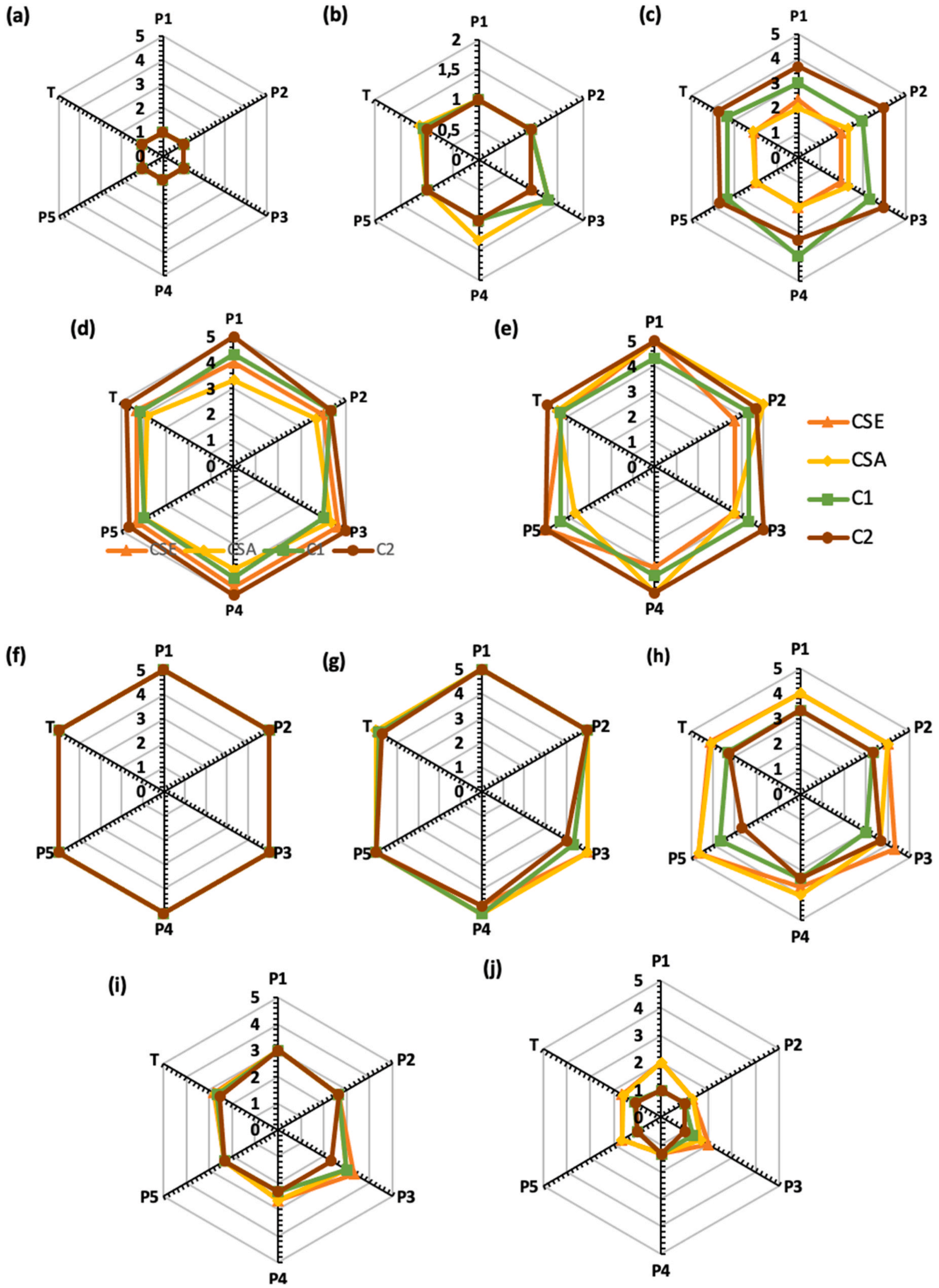

Fig. 1. Sensorial characteristics of CSE, CSA, C1 and C2 samples. Scores (1–5) are means of three evaluations of five panellists. Where: (a), (b), (c), (d) and (e): Off-Odor parameter of samples according to storage time (Day 0, Day 3, Day 5, Day 8 and Day 13), respectively. Where: (f), (g), (h), (i) and (j): Overall acceptability parameter of samples according to storage time (Day 0, Day 3, Day 5, Day 8 and Day 13), respectively.

Table 1

Instrumental color (*L**, a*, b*) and total color change ΔE during refrigerated storage (4 °C) of FAS fillets*.

Parameters	Treatments	Storage time				
		Day 0	Day 3	Day 5	Day 8	Day 13
L*	C1	49.55 ± 1.73^{Aa}	49.48 ± 0.25^{Aa}	50.85 ± 1.29^{Aa}	53.19 ± 1.72^{Ab}	53.76 ± 1.82^{Ab}
	C2	49.55 ± 1.73^{Aa}	49.75 ± 0.47^{Aa}	52.29 ± 1.40^{Ab}	52.74 ± 1.79^{Ab}	53.97 ± 1.46^{Ab}
	CSA	49.55 ± 1.73^{Aa}	49.79 ± 1.74^{Aa}	50.63 ± 1.80^{Aab}	52.45 ± 1.16^{Abc}	53.68 ± 1.61^{Ac}
	CSE	49.55 ± 1.73^{Aa}	49.81 ± 1.16^{Aa}	50.73 ± 1.62^{Aa}	52.74 ± 1.79^{Ab}	53.03 ± 1.54^{Ab}
a*	C1	12.43 ± 1.52^{Ab}	12.23 ± 0.44^{ABab}	11.97 ± 1.13^{Aab}	11.92 ± 0.81^{Aab}	11.06 ± 0.9^{Aa}
	C2	12.43 ± 1.52^{Aa}	12.01 ± 0.58^{Aa}	12.60 ± 1.49^{Aa}	11.51 ± 1.06^{Aa}	11.29 ± 1.20^{Aa}
	CSA	12.43 ± 1.52^{Aa}	12.64 ± 1.16^{ABa}	12.68 ± 1.07^{Aa}	12.19 ± 0.71^{Aa}	11.46 ± 0.41^{Aa}
	CSE	12.43 ± 1.52^{Aa}	13 ± 0.62^{Ba}	12.40 ± 0.59^{Aa}	12.20 ± 1.67^{Aa}	11.74 ± 0.72^{Aa}
b*	C1	15.41 ± 0.99^{Aa}	15.88 ± 0.47^{ABa}	16.03 ± 0.92^{ABa}	16.09 ± 0.88^{Aa}	16.37 ± 0.72^{Aa}
	C2	15.41 ± 0.99^{Aa}	15.82 ± 0.93^{ABab}	15.87 ± 0.87^{ABab}	16.16 ± 1.25^{Aab}	17.01 ± 1.66^{Ab}
	CSA	15.41 ± 0.99^{Aa}	16.22 ± 0.82^{Bab}	16.40 ± 0.44^{Bab}	16.61 ± 0.79^{Ab}	16.90 ± 1.16^{Ab}
	CSE	15.41 ± 0.99^{Aa}	15.03 ± 0.71^{Aa}	15.30 ± 1.17^{Aa}	15.66 ± 1.08^{Aa}	15.86 ± 1.12^{Aa}
ΔE	C1	0	0.52	1.51	3.74	4.53
	C2	0	0.62	2.77	3.39	4.83
	CSA	0	0.86	1.49	2.28	4.50
	CSE	0	0.63	1.19	3.21	3.58

*Values are mean ± SD of six replicates ($n = 6$). Different uppercase letters indicate statistically significant differences between analyzed samples ($A < B < C$) (column), whereas different lowercase letters indicate statistically significant differences between storage time ($a<b < c < d$) (lines), using Post-hoc HSD Tukey test ($p < 0.05$). CSE: carob seed ethanol; CSA: carob seed acetone; C1: control 1 and C2: control 2.

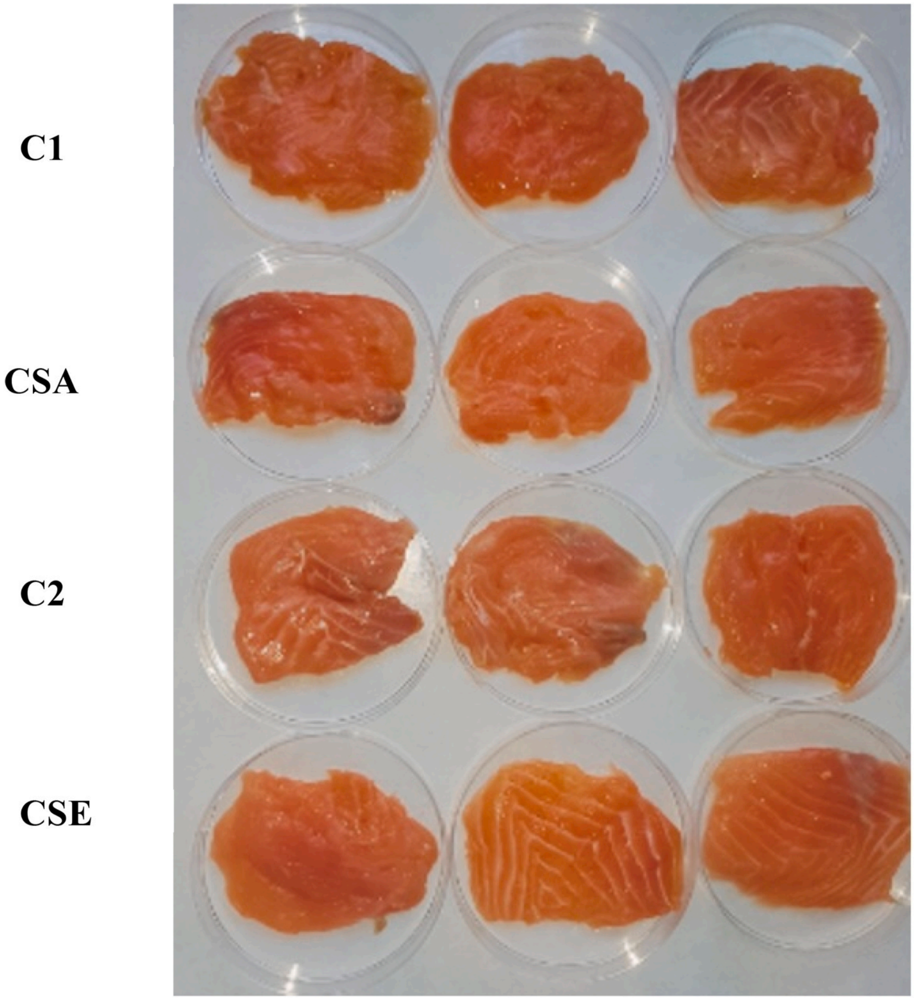

Fig. 2. Sample of Petri dish with sliced salmon fish placed in blank packaging C1 and C2 (without active agent) and active packaging consisted of CSA and CSE active film at the 5th day of experiment.

freshness and quality of these products (Freitas, Vaz-Pires, & Câmara, 2019). Values of the pH changes of FAS samples are shown in Fig. 3a. As it can be seen, the initial pH value (day 0) of fresh salmon fillet obtained in this study was 6.40 ± 0.01, similar to this obtained in the previous work of Xiong, Kamboj, Ajlouni, and Fang (2021) on the FSF with values of 6.02 ± 0.02 and 6.22, respectively. These results are within the

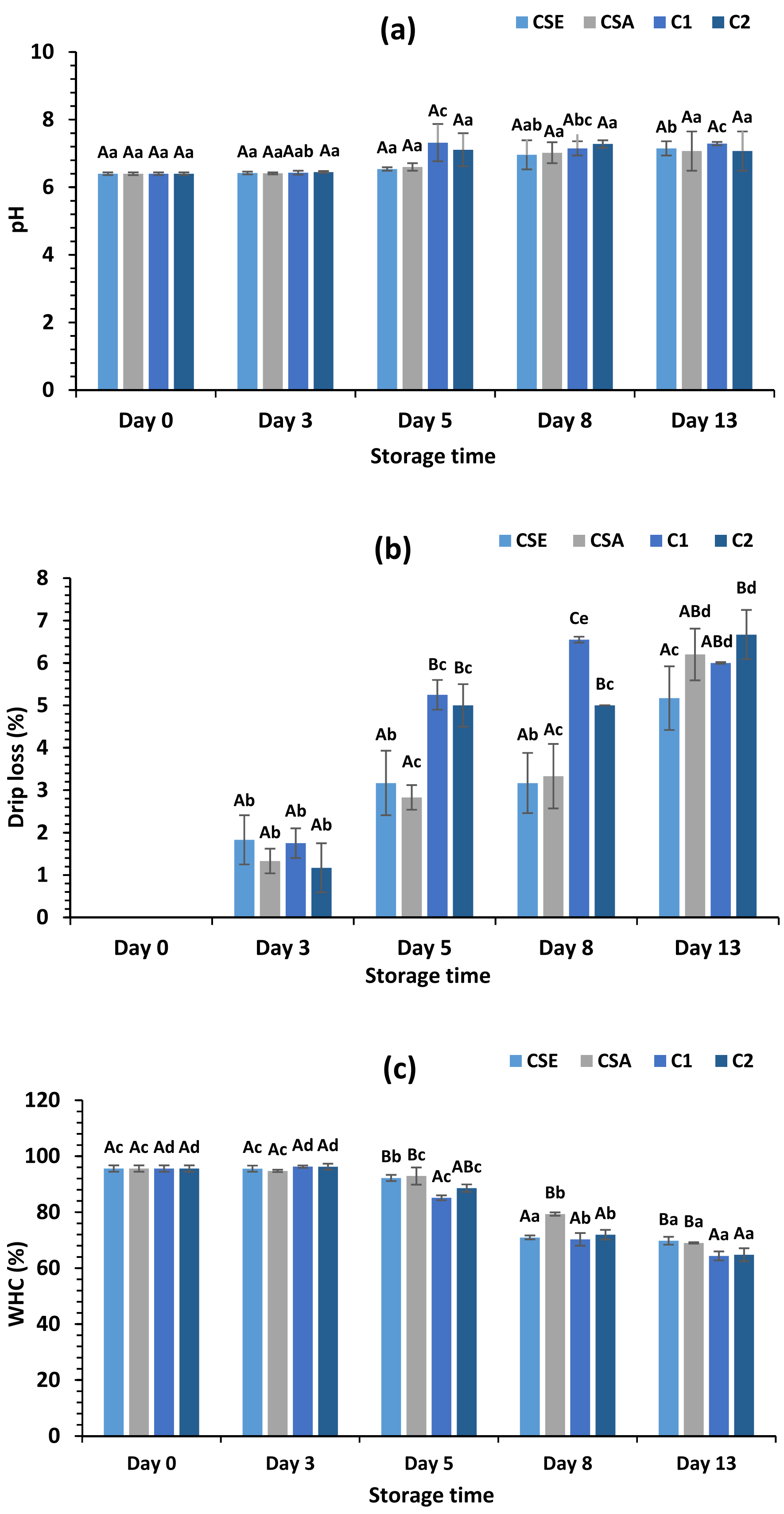

Fig. 3. Results of a) pH measurement of salmon samples at different day of storage; b) drip loss measurement of samples during salmon samples during storage time; c) WHC measurement of the different salmon samples. The results are the mean ± SD of 3 replicates (n = 3). Different uppercase letters indicate significant differences between treatments analyzed (A < B < C), whereas different lowercase letters indicate significant differences between storage time (a<b < c < d). CSE: carob seed ethanol; CSA: carob seed acetone; C1: control 1 and C2: control 2.

normal pH range for fresh salmon (~6.2) (FDA U.S. Food and Drug Administration, 2009). Throughout storage, blank samples showed a faster increase in pH values, which reached 7.32 ± 0.55 and 7.11 ± 0.49 for C1 and C2 on the 5th storage day, respectively. These results confirmed that the blank samples were degrading at a faster rate during the storage time. However, results with mean values of 6.60 ± 0.11 and 6.54 ± 0.05 were obtained in the case of samples packaged with active materials in the same storage day, which are within limits required by legislation (Júnior & de Oshiro, 2017) and validated as the threshold for fish food freshness and safety ($pH \leq 7.0$), indicating that these samples were degrading more slowly than the control samples. The reason for such difference between samples from active bio-based packaging and controls can be related to the antimicrobial action of the natural agents present in the carob seeds macerates, which inhibit the bacteria that cause protein degradation and the production of basic compounds responsible for the pH increase (Vatavali, Karakosta, Nathanailides, Georgantelis, & Kontominas, 2013). After the 5th storage day, the pH significantly increased ($p < 0.05$) in the course of the refrigerated storage for all treatments, which is associated with the production of basic amines through protein breakdown by the action of spoilage bacteria (Karabagias, Badeka, & Kontominas, 2011). It was reported that the pH increases with microbial spoilage. The reason for this is related to the formation of nitrogenous compounds such as ammonium and biogenic amines as a result of enzymatic activity and the proteolytic activity of psychrophilic bacteria (Debevere & Boskou, 1996).

3.4. Drip loss and WHC

The changes in protein-protein conformation and denaturation of important proteins, leading to the exudation from muscles of what is collected as drip loss (Fidalgo et al., 2019). The drip loss directly quantifies the loss of saleable weight and/or the deterioration of appearance and further facilitates surface microbial growth (Duun & Rustad, 2008). The result from Fig. 3b shows a significant ($p < 0.05$) increase of liquid loss through storage time. As it can be seen, samples covered with active bio-based packaging presented lower values than the control, which can be explained by the presence of antimicrobial agents in the CSE and CSA macerates that inhibit the microbial growth responsible for the structural changes in the muscle, such as proteolytic degradation of myofibrillar proteins and increased extracellular space (Kaale, Eikevik, Rustad, & Nordtvedt, 2014). Similar results were reported by Rollini et al. (2016), who demonstrated that the carvacrol-coextruded multilayer film effectively prevented microbial spoilage in fresh salmon. Another study on the valorization of carob fruit wastes to prepare bifunctional coating showed a preservative effect on salmon fillet quality by reducing bacterial growth (Goulas et al., 2019). As the storage time increased, the values increased until the last storage day and reached values of 6.00 ± 0.00%; 6.67 ± 0.58%; 5.17 ± 0.75%, and 6.20 ± 0.61% for C1, C2, CSE, and CSA, respectively, indicating the degradation of FSF samples for all treatments.

A useful tool for describing the quality of muscle foods *post-mortem* is to measure the WHC of the muscle, which is the ability of a muscle to resist water loss. It is an important quality parameter for raw Atlantic salmon, as it affects both profitability and quality by affecting the weight change during transport and storage (Lakshmanan, Parkinson, & Piggott, 2007).

In the current study, the initial mean value WHC of 95.59 ± 1.12% was found in fresh salmon, whereas values decreased significantly during refrigerated storage for all kinds of samples. However, the C1 and C2 samples showed the lowest WHC on the 13th storage day, differing significantly from CSA and CSE samples (Fig. 3c). These induced effects could be explained by the structural change in the muscle and denaturation of important myofibrillar and/or sarcoplasmic proteins. These results confirmed that the control samples were degrading at a faster rate during the storage time. However, samples covered with active bio-based packaging degraded slowly, which was demonstrated by the highest WHC. These results may be related to the active compounds of CSE and CSA macerates, such as antimicrobial agents, as previously explained, which retained better the characteristics of FSF than the control samples. A similar effect was reported elsewhere (Hultmann & Rustad, 2002; Lerfall, Bendiksen, Olsen, & Østerlie, 2015). There are indications that proteolytic enzymes, possibly originating from bacteria, are responsible for the postmortal degradation of extracellular matrix components and influence the WHC (Olsson, Seppola, & Olsen, 2007).

3.5. TBARS

TBARS is a measure of MDA, which is a good indicator for determining the progress of lipid oxidation and carbonyl and aldehyde production (Azizi-Lalabadi, Rafiei, Divband, & Ehsani, 2020). An external calibration curve was carried out to quantify the MDA in the range between 0.1 and 0.8 μg/g of MDA solution.

The TBA values for all salmon slices samples are shown in Fig. 4a. The initial value of TBA was 0.02 ± 0.03 μg MDA/g of salmon. In general, the results showed a significant *($p \leq 0.05$)* increase of TBA values for all samples after the 3rd storage day, where at the 5th day, the rate of oxidation of samples packaged with CSE and CSA active films (0.056 ± 0.033 μg MDA/g of salmon and 0.088 ± 0.054 μg MDA/g of salmon, respectively) were lower than the corresponding values of the control group, reaching values of 0.188 ± 0.011 μg MDA/g of salmon and 0.166 ± 0.027 μg MDA/g of salmon for C1 and C2, respectively. Obtained results may be attributed to the increased oxidation of unsaturated fatty acids and partial dehydration of the salmon fillet. The difference of obtained results between control and active samples is mainly due to the antioxidant activity of carob seeds macerates, based on natural antioxidant compounds immobilized in the multilayer biomaterial (Ait Ouahioune et al., 2022). The mechanism of antioxidant protection that active compounds from carob seeds macerates offer to FSF does not imply a positive migration of antioxidants to salmon but a real scavenging and non-migrating system that can take place without direct contact between the carob seeds macerates and the fresh salmon (Carrizo, Taborda, Nerín, & Bosetti, 2015; Vera et al., 2016; Wrona, Bentayeb, & Nerín, 2015).

This new kind of active packaging works differently than those usually conceived as the material supplying antioxidants or acting as oxygen absorbers. At the same time, it's a concept of a free radical scavenger (Carrizo et al., 2015). The oxidation process is a reaction of radicals initiated by OH· and O· free radicals. When they are removed, the reaction does not take place, as it was demonstrated in the previous works of Pezo, Salafranca, and Nerín (2006); Pezo et al. (2008); Colon and Nerin (2015); Carrizo et al. (2015); Nerín (2011); Dicastillo et al. (2011). The free radicals have a short life, as they are very reactive, and can easily permeate through the polymers. Thus, they will arrive at the bioactive compounds positions, where they were grafted in the adhesive formulate and trapped by the carob seeds molecules endowed with antioxidant properties as a free radical scavenger and therefore protects the FSF versus oxidation processes (Moudache, Nerin, Colon, & Zaidi, 2017). There are a few studies regarding the active antioxidant packaging formulated with natural antioxidants from the carob (Ait Ouahioune et al., 2022; Goulas et al., 2019). It was reported that the packaged salmon with carob polyphenolic coating retains the quality of salmon by reducing lipid oxidation, avoiding the presence of undesirable off-flavors in salmon fillets during refrigerated storage at 6 °C (Goulas et al., 2019). According to Castro, Andrade, Silva, Vaz, and Vilarinho (2019), the TBA values of 0.5 μg MDA/g are the acceptable levels of MDA in the fish flesh, which normally correspond to a development of an objectionable odor. Therefore, the TBA values of our samples covered with CSE and CSA active films during all the storage period was lower than that allowed for the fresh fish for the perception of lipid oxidation, except for the C1 on the last storage day. Thus, these findings indicate that both active bio-based films incorporated with the carob seeds macerates, considered agro-food waste, had the most

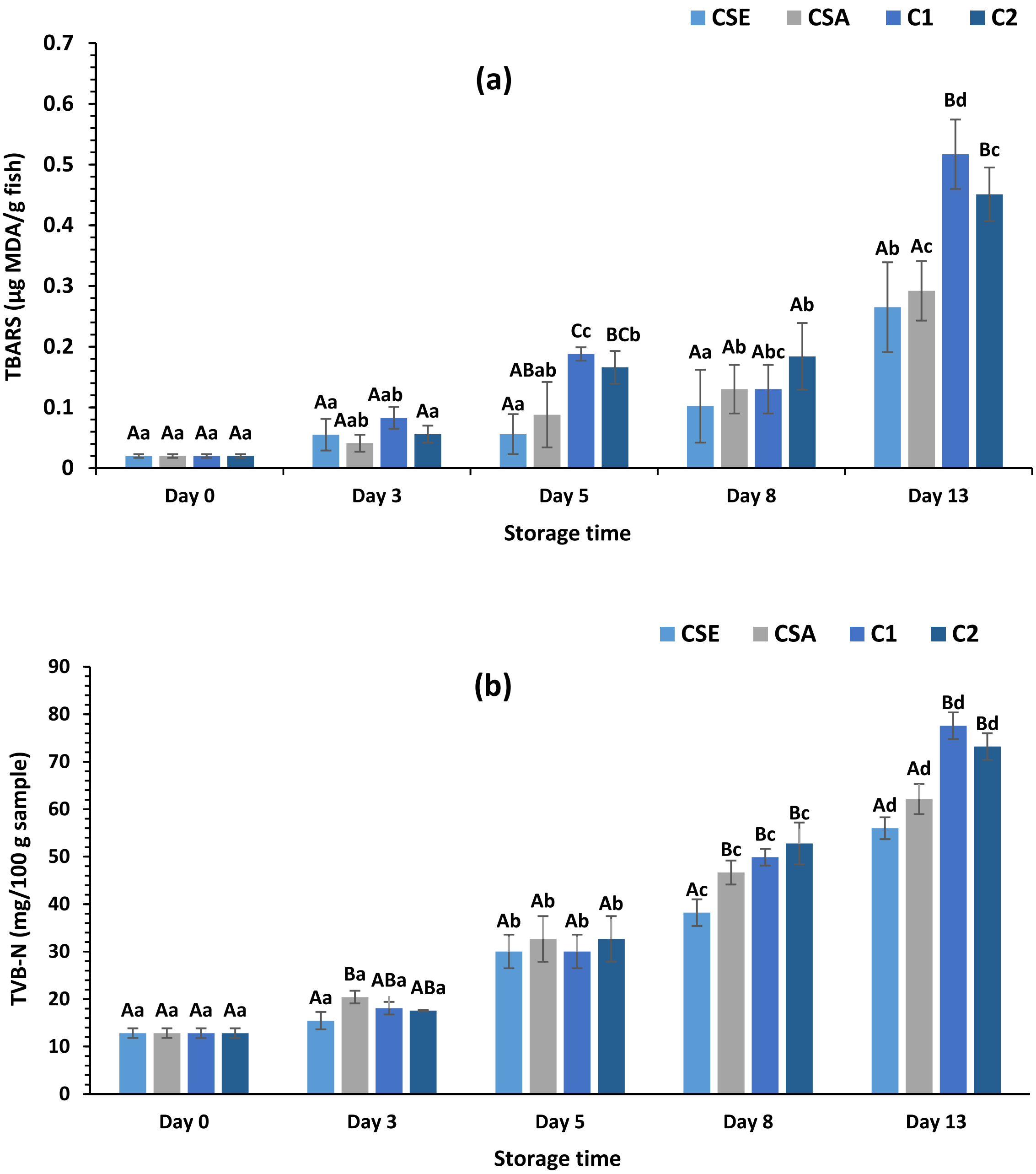

Fig. 4. Results of a) Concentration of thiobarbituric acid (TBA) (mg of malondialdehyde/100 g of sample) in the different salmon samples (*Salmo salar* L.) stored at 4 °C; b) TVB-N values for salmon samples during 14 days of storage at 5 °C. The results are the mean ± SD of three replicates (n = 3). Different uppercase letters indicate statistically significant differences between samples analyzed (A < B < C) (column), whereas different lowercase letters indicate statistically significant differences between storage time (a<b < c < d) (lines), using Post-hoc HSD Tukey test ($p < 0.05$). CSE: carob seed ethanol; CSA: carob seed acetone; C1: control 1 and C2: control 2.

protective effect on the delay of fat oxidation in the FSF. Moreover, they could also be suitable for packaging other types of food such as meat. Nevertheless, scale-up tests regarding this type of packaging are mandatory to assess its commercial and economic viability and add value to several industries' by-products.

3.6. TVB-N

TVB-N is one of the most widely used indices of seafood quality. It is a term that includes measurement of trimethylamine, dimethylamine, ammonia, and other compounds associated with seafood spoilage, which increases as spoilage progresses (Mohan, Ravishankar, Lalitha, & Srinivasa Gopal, 2012). Fig. 4b shows the evolution of TVB-N values versus the storage period. As can be seen at the beginning of storage, FSF showed a TVB-N value of 12.83 ± 1.01 mg N/100 g of salmon, which is slightly higher than that reported by Alves et al. (2018). The values of TVB-N increased significantly ($p < 0.05$) throughout the storage time for all studied samples, showing on day 13 values of 77.59 ± 2.81; 73.21 ± 2.81; 56 ± 2.31 and 62.13 ± 3.16 mg N/100 g of salmon for C1, C2, CSE, and CSA samples, respectively. TVB-N increase is related to spoilage by bacteria and the activity of endogenous enzymes. CSE and CSA active biopackaging showed lower values than the control samples, mainly between the 3rd and 5th storage days. Reasons for these differences include the enhanced microbial load or the ability of the carob seeds extract to de-aminate non-protein nitrogen compounds (Ojagh, Núñez-Flores, López-Caballero, Montero, & Gómez-Guillén, 2011).

High TVB-N values are undesirable since they indicate that salmon fillets are spoiled. Based on the sensory attributes of the current study, the more realistic TVB-N limit of 35 mg N/100 g of fish was proposed as the upper limit above, which the salmon is considered spoiled and not suitable for human consumption and within the value that was also established by Regulation (EC) No 2074/2005. As can be seen, a significant increase in the TVB-N values of the different samples after the 5th storage day was observed (Fig. 4b), exceeding the maximum limit of acceptability, indicating a good correlation with the sensory analysis results.

3.7. Correlation matrix and multivariate statistical analysis

Table S1 in supplementary material shows the correlation matrix among the different parameters used to evaluate the FSF quality. Different values corresponded to the discrimination indexes between parameters. The results showed a highly significant correlation between pH, TBA, TVB-N, WHC, sensory attributes, and L^* parameters, whereas a significant correlation with a^* and b^* parameters was obtained.

Multivariate statistical analysis was applied to compare C1, C2, CSE, and CSA salmon samples stored. The technique most used is PCA, which allows the reduction of a large set of multivariate data into a small number of principal components and gives a simplified interpretation of data. In the current study, the observations are the different samples of packaged FSF, and the variables are the different parameters used to evaluate the sample quality. The PCA score biplot with the projection of the individuals on the factorial plane (1 × 2) and projection of the variables on the factorial plane (1 × 2) based on storage time are displayed in Fig. 5a and b, respectively.

As shown in Fig. 5b, the correlation between the results of the different parameters used in the salmon quality assessment was obtained, agreeing with the results of the correlation matrix. PC1 and PC2 (85.21% and 5.65%, respectively) explained 90.86% of the total variation. Thus, they can be used as an adequate explanation of the data.

In total, 10 standardized variables were introduced to create the covariance matrix. The first principal component (PC1) that represented 85.21% of the variance was found to be highly correlated with all the variables, with a positive correlation coefficient for drip loss (0.931), pH (0.909), TBA (0,901), TVB-N (0.979), off-odor (0.956), L^* (0.969) and b^* (0.756), whereas negative with WHC (−0.961), overall acceptability (−0.990) and a^* (−0.853). The PC2 (5.65% of the variance) had a positive correlation coefficient with drip loss (0.165), pH (0.286), off-odor (0.231), L^* (0.143) and a^* (0.287) and negative with TBA (−0.233), TVB-N (−0.084), WHC (−0.095), overall acceptability (−0.034) and b^* (−0.478). Some of the variables are highly correlated and appeared together in the biplot (Fig. 5b). At the right, pH, off-odor drip loss, TVB-N, TBA and b^*. At the left, are overall acceptability, WHC and a^*.

Four groups were observed in the score plot (Fig. 5a). As it can be seen, it is possible to observe that all the clusters appear distinct, but, in some cases, there is not a clear separation between the samples of the same cluster. The CSAD3, CSAD5, C1D3, C2D3 emerged into a cluster with the samples of day 0 showing the similar characteristic of FAS. CSAD5 and CSED5 samples were clustered with samples of day 0 and samples of day 3, respectively. This phenomenon can be explained by the effectiveness of the active biopackaging incorporated with CSA and CSE macerates in the deterioration delay of the FAS compared with C1D5 and C2D5, which degraded at a faster rate (clustered together C1D8, C2D8, CSED8, CSED13). Along with the storage time, CSED13 and CSAD8 emerged with samples of day 8 and day 13, respectively, showing that samples from CSE retain better the characteristic of salmon samples than CSA on the last storage day. The results of PCA demonstrated that there was some useful information on the effect of active biopackaging on the shelf-life extension of salmon samples compared to the control samples.

4. Conclusions

A new antioxidant cellulose biofilm based on the incorporation of carob seeds macerates was successfully developed. This study reports the beneficial effect of CSA and CSE biopackaging on FSF samples stored at 4 ± 1 °C, especially in maintaining the color values closer to those of fresh salmon at the beginning of storage and keeping salmon samples with lower pH, TBA, TVB-N, drip losses values, and higher WHC values. Moreover, satisfactory sensory evaluation results were obtained with samples acceptable between the 3rd and 5th days compared to the control samples. In conclusion, the developed active biopackaging can scavenge the free radicals and inhibit lipids and proteins' oxidation. It is

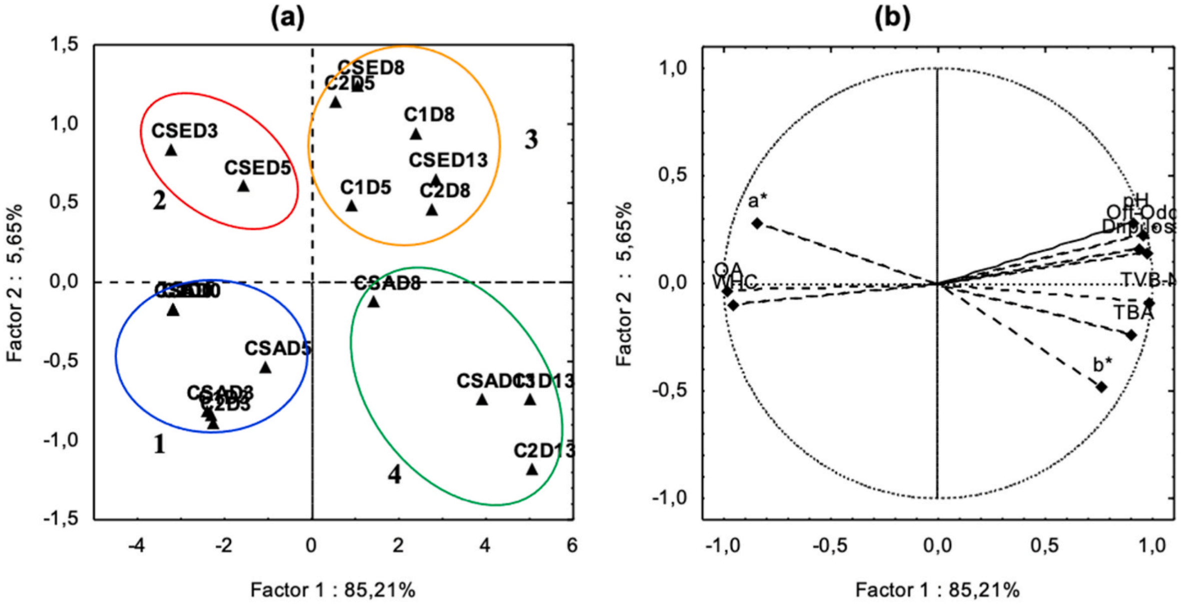

Fig. 5. Principal component analysis (PCA) biplot; (a): projection of the individuals on the factorial plane (1 × 2). (b): projection of the variables on the factorial plane (1 × 2). CSE: carob seed ethanol; CSA: carob seed acetone; C1: control 1 and C2: control 2. D1; D3; D5; D8 and D13: storage days.

an effective, promising, and efficient method for increasing the shelf-life quality of fresh salmon fish, suggesting its application in the food industry. It is considered a sustainable approach to synthetic plastic material, allowing an added value to the wastes of food industries.

CRediT authorship contribution statement

Lidia Ait Ouahioune: Methodology, Conceptualization, Validation, Investigation, Data curation, Writing – original draft, Writing – review & editing. **Magdalena Wrona:** Conceptualization, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review & editing, Supervision. **Cristina Nerín:** Conceptualization, Resources, Writing – review & editing, Supervision, Project administration, Funding acquisition. **Djamel Djenane:** Conceptualization, Resources, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2021.113015.

References

Abidar, S., Boiangiu, R. S., Dumitru, G., Todirascu-Ciornea, E., Amakran, A., Cioanca, O., et al. (2020). The aqueous extract from ceratonia siliqua leaves protects against 6-hydroxydopamine in zebrafish: Understanding the underlying mechanism. *Antioxidants, 9*(4), 304. https://doi.org/10.3390/antiox9040304

Adilah, A. N., Jamilah, B., Noranizan, M. A., & Hanani, Z. A. N. (2018). Utilization of mango peel extracts on the biodegradable films for active packaging. *Food Packaging and Shelf Life, 16*, 1–7. https://doi.org/10.1016/j.fpsl.2018.01.006, 2018.

Ait Ouahioune, L., Wrona, M., Becerril, R., Salafranca, J., Nerin, C., & Djenane, D. (2022). Ceratonia siliqua L. kibbles, seeds and leaves as a source of volatile bioactive compounds for antioxidant food biopackaging applications. *Food Packaging and Shelf Life, 31*. https://doi.org/10.1016/j.fpsl.2021.100764, 100764.

Alves, V. L. C. D., Rico, B. P. M., Cruz, R. M. S., Vicente, A. A., Khmelinskii, I., & Vieira, M. C. (2018). Preparation and characterization of a chitosan film with grape seed extract-carvacrol microcapsules and its effect on the shelf-life of refrigerated Salmon (Salmo salar). *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology, 89*, 525–534. https://doi.org/10.1016/j.lwt.2017.11.013, 2018.

Azizi-Lalabadi, M., Rafiei, L., Divband, B., & Ehsani, A. (2020). Active packaging for Salmon stored at refrigerator with Polypropylene nanocomposites containing 4A zeolite, ZnO nanoparticles, and green tea extract. *Food Sciences and Nutrition, 8*(12), 6445–6456. https://doi.org/10.1002/fsn3.1934

Bell, J. G., Henderson, R. J., Tocher, D. R., & Sargent, J. R. (2004). Replacement of dietary fish oil with increasing linseed oil levels: Modify flesh fatty acid compositions in atlantic salmon (Salmo salar) using a fish oil finishing diet. *Lipids, 39*(3), 223–232. https://doi.org/10.1007/s11745-004-1223-5

Ben Ayache, S., Behija Saafi, E., Emhemmed, F., Flamini, G., Achour, L., & Muller, C. D. (2020). Biological activities of aqueous extracts from carob plant (ceratonia siliqua L.) by antioxidant, analgesic and proapoptotic properties evaluation. *Molecules, 25*(14), 3120. https://doi.org/10.3390/molecules25143120

Cao, T. L., & Song, K. B. (2020). Development of bioactive Bombacaceae gum films containing cinnamon leaf essential oil and their application in packaging of fresh salmon fillets. *Lebensmittel-Wissenschaft & Technologie, 131*, 1–8. https://doi.org/10.1016/j.lwt.2020.109647, 109647.

Carrizo, D., Taborda, G., Nerín, C., & Bosetti, O. (2015). Extension of shelf life of two fatty foods using a new antioxidant multilayer packaging containing green tea extract. *Innovative Food Science And Emerging Technologies, 33*, 534–541. https://doi.org/10.1016/j.ifset.2015.10.018

Castro, F. V. R., Andrade, M. A., Silva, A. S., Vaz, M. F., & Vilarinho, F. (2019). The contribution of a whey protein film incorporated with green tea extract to minimize the lipid oxidation of salmon (Salmo salar L.). *Foods, 8*(8), 327. https://doi.org/10.3390/foods8080327

Colon, M., & Nerin, C. (2015). Development and application of an analytical procedure for specific migration of green tea compounds in IV gamma nectarine active packaging. *Food Control, 57*, 419–425. https://doi.org/10.1016/j.foodcont.2015.04.035

Debevere, J., & Boskou, G. (1996). Effect of modified atmosphere packaging on the TVB/TMA-producing microflora of cod fillets. *International Journal of Food Microbiology, 31*(1–3), 221–229. https://doi.org/10.1016/0168-1605(96)01001-X

Dicastillo, C. L., Nerín, C., Alfaro, P., Catalá, R., Gavara, R., & Hernandez-Muñoz, P. (2011). Development of new antioxidant active packaging films base on ethylene vinyl alcohol copolymer (EVOH) and green tea extract. *Journal of Agricultural and Food Chemistry, 59*, 7832–7840. https://doi.org/10.1021/jf201246g

Djebari, S., Wrona, M., Boudria, A., Salafranca, J., Nerin, C., Bedjaoui, K., et al. (2021). Study of bioactive volatile compounds from different parts of Pistacia lentiscus L. extracts and their antioxidant and antibacterial activities for new active packaging application. *Food Control, 120*, 1–8. https://doi.org/10.1016/j.foodcont.2020.107514, 107514.

Djenane, D. (2015). Chemical profile, antibacterial and antioxidant activity of algerian citrus essential oils and their application in sardina pilchardus. *Foods, 4*(2), 208–228. https://doi.org/10.3390/foods4020208

Djenane, D., Aboudaou, M., Ferhat, M. A., Ouelhadj, A., & Ariño, A. (2019). Effect of the aromatisation with summer savory (Satureja hortensis L.) essential oil on the oxidative and microbial stabilities of liquid whole eggs during storage. *Journal of Essential Oil Research, 31*(5). https://doi.org/10.1080/10412905.2019.1610516

Djenane, D., Sánchez-Escalante, A., Beltrán, J. A., & Roncalés, P. (2001). Extension of the retail display life of fresh beef packaged in modified atmosphere by varying lighting conditions. *Journal of Food Science, 66*(1), 181–186. https://doi.org/10.1111/j.1365-2621.2001.tb15603.x

Duun, A. S., & Rustad, T. (2008). Quality of superchilled vacuum packed Atlantic salmon (Salmo salar) fillets stored at _1.4 and _3.6 _C. *Food Chemistry, 106*(1), 122e131.

FDA U.S. Food and Drug Administration. (2009). CFSAN—bad bug book—pH values of various foods. Available from: www.fda.gov. (Accessed 29 June 2009).

Fernández, K., Aspe, E., & Roeckel, M. (2009). Shelf-life extension on fillets of Atlantic Salmon (*Salmo salar*) using natural additives, superchilling and modified atmosphere packaging. *Food Control, 20*(11), 1036–1042. https://doi.org/10.1016/j.foodcont.2008.12.010

Fidalgo, L. G., Castro, R., Trigo, M., Aubourg, S. P., Delgadillo, I., & Saraiva, J. A. (2019). Quality of fresh atlantic salmon (Salmo salar) under hyperbaric storage at low temperature by evaluation of microbial and physicochemical quality indicators. *Food and Bioprocess Technology*. https://doi.org/10.1007/s11947-019-02346-3

Fidan, H., Stankov, S., Petkova, N., Petkova, Z., Iliev, A., Stoyanova, M., et al. (2020). Evaluation of chemical composition, antioxidant potential and *functional* properties of carob (Ceratonia siliqua L.) seeds. *Journal of Food Science & Technology, 57*(7), 2404–2413. https://doi.org/10.1007/s13197-020-04274-z

Freitas, J., Vaz-Pires, P., & Câmara, J. S. (2019). Freshness assessment and shelf-life prediction for Seriola dumerili from aquaculture based on the quality index method. *Molecules, 24*(19), 3530. https://doi.org/10.3390/molecules24193530

Ghaly, A. E., Dave, D., Budge, S., & Brooks, M. S. (2010). Fish spoilage mechanisms and preservation techniques: Review. *American Journal of Applied Sciences, 7*, 846–864. https://doi.org/10.3844/ajassp.2010.859.877

Giménez, B., Roncalés, P., & Beltrán, J. A. (2005). The effects of natural antioxidants and lighting conditions on the quality of salmon (Salmo salar) fillets packaged in modified atmosphere. *Journal of the Science of Food and Agriculture, 85*(6), 1033–1040. https://doi.org/10.1002/jsfa.2055

Goulas, V., Hadjivasileiou, L., Primikyri, A., Michael, C., Botsaris, G., Tzakos, A. G., et al. (2019). Valorization of carob fruit residues for the preparation of novel bi-functional polyphenolic coating for food packaging applications. *Molecules, 24*(17), 1–10. https://doi.org/10.3390/molecules24173162

Guyon, C., Meynier, A., & de Lamballerie, M. (2016). Protein and lipid oxidation in meat: A review with emphasis on high-pressure treatments. *Trends in Food Science & Technology, 50*, 131–143. https://doi.org/10.1016/j.tifs.2016.01.026

Hultmann, L., & Rustad, T. (2002). Textural changes during iced storage of salmon (Salmo salar) and cod (Gadus morhua). *Journal of Aquatic Food Product Technology, 11*(3/4), 105e123. https://doi.org/10.1300/J030v11n03_09

ISO-6496. (1999). No Determination of moisture and other volatile matter content. In *Animal feeding stuffs*.

Júnior, J. M., & de Oshiro, M. (2017). Atualizações importantes introduzidas pelo novo Regulamento de Inspeção Industrial e Sanitária de Produtos de Origem Animal: Decreto n° 9.013 de 29 de março de 2017. *Vigilância Sanitária em Debate. 5. Vigilância Sanitária em Debate*, 73–80. https://doi.org/10.22239/2317-269x.01019

Kaale, L. D., Eikevik, T. M., Rustad, T., & Nordtvedt, T. S. (2014). Changes in water holding capacity and drip loss of Atlantic salmon (Salmo salar) muscle during superchilled storage. *LWT – Food Science and Technology, 55*, 528–535. https://doi.org/10.1016/j.lwt.2013.10.021

Karabagias, I., Badeka, A., & Kontominas, M. G. (2011). Shelf life extension of lamb meat using thyme or orégano essential oils and modified atmosphere packaging. *Meat Science, 88*, 109–116. https://doi.org/10.1016/j.meatsci.2010.12.010

Khwaldia, K., Ferez, C., Banon, S., Desobry, S., & Hardy, J. (2004). Milk proteins for edible films and coatings. *Critical Reviews in Food Science and Nutrition, 44*(4), 239–251. https://doi.org/10.1080/10408690490464906

Lakshmanan, R., Parkinson, J. A., & Piggott, J. R. (2007). High-pressure processing and water-holding capacity of fresh and cold-smoked salmon (Salmo salar). *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology, 40*(3), 544–551. https://doi.org/10.1016/j.lwt.2005.12.003

Lammi, S., Barakat, A., Mayer-Laigle, C., Djenane, D., Gontard, N., & Angellier-Coussy, H. (2017). Dry fractionation of olive pomace as a sustainable process to produce fillers for biocomposites. *Powder Technology, 326*, 44–53. https://doi.org/10.1016/j.powtec.2017.11.060, 2018.

Lammi, S., Le Moigne, N., Djenane, D., Gontard, N., & Angellier-Coussy, H. (2018). Dry fractionation of olive pomace for the development of food packaging biocomposites. *Industrial Crops and Products, 120*, 250–261. https://doi.org/10.1016/j.indcrop.2018.04.052, 2018.

Lan, W., Wang, S., Zhang, Z., Liang, X., Liu, X., & Zhang, J. (2021). Development of red apple pomace extract/chitosan-based films reinforced by TiO2 nanoparticles as a multifunctional packaging material. *International Journal of Biological Macromolecules, 168*, 105–115. https://doi.org/10.1016/j.ijbiomac.2020.12.051, 2021.

Lerfall, J., Bendiksen, E.Å., Olsen, J. V., & Østerlie, M. (2016). A comparative study of organic- versus conventional Atlantic salmon. II. Fillet color, carotenoid- and fatty acid composition as affected by dry salting, cold smoking and storage. *Aquaculture, 451*, 369–376. https://doi.org/10.1016/j.aquaculture.2015.10.004

Lerfall, J., Roth, B., Skare, E. F., Henriksen, A., Betten, T., Dziatkowiak-Stefaniak, M. A., et al. (2015). Pre-mortem stress and the subsequent effect on flesh quality of pre-rigor filleted Atlantic salmon *(Salmo salar L.)* during ice storage. *Food Chemistry, 175*, 157–165. https://doi.org/10.1016/j.foodchem.2014.11.111

Merlo, T. C., Contreras-Castillo, C. J., Saldaña, E., Barancelli, G. V., Dargelio, M. D. B., Yoshida, C. M. P., et al. (2019). Incorporation of pink pepper residue extract into chitosan film combined with a modified atmosphere packaging: Effects on the shelf life of salmon fillets. *Food Research International, 125*, 1–10. https://doi.org/10.1016/j.foodres.2019.108633, 2019.

Mohan, C. O., Ravishankar, C. N., Lalitha, K. V., & Srinivasa Gopal, T. K. (2012). Effect of chitosan edible coating on the quality of double filleted Indian oil sardine (Sardinella longiceps) during chilled storage. *Food Hydrocolloids, 26*(1), 167–174. https://doi.org/10.1016/j.foodhyd.2011.05.005

Moudache, M., Nerin, C., Colon, M., & Zaidi, F. (2017). Antioxidant effect of an innovative active plastic film containing olive leaves extract on fresh pork meat and its evaluation by Raman spectroscopy. *Food Chemistry, 229*, 98–103. https://doi.org/10.1016/j.foodchem.2016.06.001

Nerín, C. (2011). Antioxidant active packaging and antioxidant edible films Chapter 31 in the book "Oxidation in foods and beverages and antioxidant applications". In E. A. Decker, R. J. Elias, & D. J. McClements (Eds.), *Oxidation in foods and beverages and antioxidant applications* (1st ed., pp. 496–515). Cambridge, United Kingdom: Woodhead Publishing. Management in Different Industry Sectors, 2.

Nerín, C., Aznar, M., & Carrizo, D. (2016). Food contamination during food process. *Trends in Food Science & Technology, 48*, 63–68. https://doi.org/10.1016/j.tifs.2015.12.004

Ojagh, S. M., Núñez-Flores, R., López-Caballero, M. E., Montero, M. P., & Gómez-Guillén, M. C. (2011). Lessening of high-pressure-induced changes in Atlantic salmon muscle by the combined use of a fish gelatin-lignin film. *Food Chemistry, 125* (2), 595–606. https://doi.org/10.1016/j.foodchem.2010.08.072

Olsson, G. B., Seppola, M. A., & Olsen, R. L. (2007). Water-holding capacity of wild and farmed cod (Gadus morhua) and haddock (Melanogrammus aeglefinus) muscle during ice storage. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology, 40*(5), 793–799. https://doi.org/10.1016/j.lwt.2006.04.004

Pezo, D., Salafranca, J., & Nerín, C. (2006). Design of a method for generation of gas-phase hydroxyl radicals, and use of HPLC with fluorescence detection to assess the antioxidant capacity of natural essential oils. *Analytical and Bioanalytical Chemistry, 385*, 1241–1246. https://doi.org/10.1007/s00216-006-0395-4

Pezo, D., Salafranca, J., & Nerín, C. (2008). Determination of the antioxidant capacity of active food packagings by in situ gas-phase hydroxyl radical generation and high-performance liquid chromatography–fluorescence detection. *Journal of Chromatography A, 1178*(1–2), 126–133. https://doi.org/10.1016/j.chroma.2007.11.062

Quezel, P., & Santa, S. (1963). *Nouvelle flore de l'Algérie et des régions désertiques méridionales (tome 1) (Editions d)*.

Quiles-Carrillo, L., Mellinas, C., Garrigos, M. C., Balart, R., & Torres-Giner, S. (2019). Optimization of microwave-assisted extraction of phenolic compounds with antioxidant activity from carob pods. *Food Analytical Methods. Food Analytical Methods*. https://doi.org/10.1007/s12161-019-01596-3

Regulation (EC) No 2074/2005. Official Journal of the European union, 27–59.

Rollini, M., Nielsen, T., Musatti, A., Limbo, S., Piergiovanni, L., Hernandez Munoz, P., et al. (2016). Antimicrobial performance of two different packaging materials on the microbiological quality of fresh salmon. *Coatings, 6*(1), 6. https://doi.org/10.3390/coatings6010006

Ruff, N., FitzGerald, R. D., Cross, T. F., & Kerry, J. P. (2002). Fillet shelf-life of atlantic halibut Hippoglossus hippoglossus L. fed elevated levels of α-tocopheryl acetate. *Aquaculture Research, 33*(13), 1059–1071. https://doi.org/10.1046/j.1365-2109.2002.00770.x

Sallam, K. I. (2007). Chemical, sensory and shelf life evaluation of sliced salmon treated with salts of organic acids. *Food Chemistry, 101*(2), 592–600. https://doi.org/10.1016/j.foodchem.2006.02.019

Santonocito, D., Granata, G., Geraci, C., Panico, A., Siciliano, E. A., Raciti, G., et al. (2020). Carob seeds: Food waste or source of bioactive compounds? *Pharmaceutics, 12*(11), 1–12. https://doi.org/10.3390/pharmaceutics12111090

Shahidi, F., & Botta, J. R. (1994). Seafoods: Chemistry, processing technology and quality. *In Seafoods: Chemistry, Processing Technology and Quality*. https://doi.org/10.1007/978-1-4615-2181-5

Soares, N., Silva, P., Barbosa, C., Pinheiro, R., & Vicente, A. A. (2017). Comparing the effects of glazing and chitosan-based coating applied on frozen salmon on its organoleptic and physicochemical characteristics over six-months storage. *Journal of Food Engineering, 194*, 79–86. https://doi.org/10.1016/j.jfoodeng.2016.07.021, 2017.

Tiwari, B. K., Muthukumarappan, K., O'Donnell, C. P., & Cullen, P. J. (2008). Kinetics of freshly squeezed orange juice quality changes during ozone processing. *Journal of Agricultural and Food Chemistry, 56*(15), 6416–6422. https://doi.org/10.1021/jf800515e

Union Europea. (2011). In *, 21. Commission Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food Offical Journal of the European Union* (pp. 1–136).

Van Haute, S., Raes, K., Devlieghere, F., & Sampers, I. (2017). Combined use of cinnamon essential oil and MAP/vacuum packaging to increase the microbial and sensorial shelf life of lean pork and salmon. *Food Packaging and Shelf Life, 12*, 51–58. https://doi.org/10.1016/j.fpsl.2017.02.004

Vatavali, K., Karakosta, L., Nathanailides, C., Georgantelis, D., & Kontominas, M. G. (2013). Combined effect of chitosan and oregano essential oil dip on the microbiological, chemical, and sensory attributes of red porgy (*Pagrus pagrus*) stored in ice. *Food and Bioprocess Technology, 6*, 3510–3521. https://doi.org/10.1007/s11947-012-1034-z

Vera, P., Echegoyen, Y., Canellas, E., Nerin, C., Palomo, M., Madrid, Y., et al. (2016). Nano selenium as antioxidant agent in a multilayer food packaging material. *Analytical and Bioanalytical Chemistry*, 1–12. https://doi.org/10.1007/s00216-016-9780-9

Wrona, M., Bentayeb, K., & Nerín, C. (2015). A novel active packaging for extending the shelf-life of fresh mushrooms (*Agaricus bisporus*). *Food Control, 54*, 200–207. https://doi.org/10.1016/j.foodcont.2015.02.008

Wrona, M., Nerin, C., Alfonso, M. J., & Caballero, M. A. (2017). Antioxidant packaging with encapsulated green tea for fresh minced meat. *Innovative Food Science & Emerging Technologies, 41*, 307–313. https://doi.org/10.1016/j.ifset.2017.04.001

Wrona, M., Vera, P., Pezo, D., & Nerín, C. (2017). Identification and quantification of odours from oxobiodegradable polyethylene oxidised under a free radical flow by headspace solid-phase microextraction followed by gas chromatography-olfactometry-mass spectrometry. *Talanta, 172*, 37–44. https://doi.org/10.1016/j.talanta.2017.05.022

Xiong, Y., Kamboj, M., Ajlouni, S., & Fang, Z. (2021). Incorporation of salmon bone gelatine with chitosan, gallic acid and clove oil as edible coating for the cold storage of fresh salmon fillet. *Food Control, 125*, 1–9. https://doi.org/10.1016/j.foodcont.2021.107994, 107994.

Yagiz, Y., Kristinsson, H. G., Balaban, M. O., Welt, B. A., Raghavan, S., & Marshall, M. R. (2010). Correlation between astaxanthin amount and a* value in fresh Atlantic salmon (Salmo salar) muscle during different irradiation doses. *Food Chemistry, 120* (1), 121–127. https://doi.org/10.1016/j.foodchem.2009.09.086

Zhao, J., Xiong, Y. L., & McNear, D. H. (2013). Changes in structural characteristics of antioxidative soy protein hydrolysates resulting from scavenging of hydroxyl radicals. *Journal of Food Science, 78*(2), 152–159. https://doi.org/10.1111/1750-3841.12030