

Article

Addition of by-Products and Flavored Hydrocolloids Filling in Californian-Style Black Olive to Reduce Acrylamide Formation

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Abstract: Oxidized black table olives are frequently consumed in the Mediterranean diet. To improve food quality, the use of by-products is an emergent strategy that should be more studied. With a better understanding of the use of by-products, healthier food with the highest possible quality could be obtained, increasing its added value. Different by-products at different concentrations (1:10 and 1:100) and phenol-rich aromatized hydrocolloids were added to Californian-style black olive in order to evaluate its effect in acrylamide. In general, the addition of by-products before the sterilization process resulted in a significant reduction of the acrylamide content (10–60%). The highest inhibition was obtained with the phenolic extract at a 1:10 dilution. Furthermore, flavored olives stuffed with higher concentrations caused a greater reduction in acrylamide content. The ‘Garlic’ and ‘Thyme’ flavors showed the greatest reduction of this toxic substance. The tasting panel considered that olives with a 1:10 dilution and olives filled with flavored hydrocolloids at low concentrations had a pleasant odor and positive sensory attributes. Orange peel was the recommended by-product to mitigate the effects of acrylamide since it obtained the highest score both in acrylamide inhibition and in the taste panel. As for the hydrocolloids, ‘Thyme’ showed the greatest inhibition effect but did not perform well in the taste panel, with similar effects to ‘Oregano’ and ‘Garlic.’ The current research attempts to underline the use of local by-products to obtain additives rich in antioxidant activity that could enhance oxidized black table olives, becoming a healthier, safer and better-quality products. Moreover, this connects with the idea of moving from a linear economy to a circular economy, obtaining a product of high economic value from a by-product.

Keywords: by-products; flavored hydrocolloid; acrylamide; table olive; sensory analysis



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1. Introduction

In the last decades, the production of food, and therefore, the production of food waste have been increasing sharply. This is a huge environmental problem and a global concern, linked to the slow development of effective strategies to manage, control and process these residues [1]. However, food waste valorization has been studied because of its content in valuable compounds, such as nutrients (e.g., carbohydrates, proteins, or lipids), bioactive compounds (e.g., polyphenols, vitamins, among others) and other compounds that could be used in several industrial sectors [1–3].

Some of the main relevant industries worldwide are related to the production of wine, olive oil, table olives or juices, and it has become necessary to study how to control their

residues. In the case of the wine industries, different studies have been performed using waste during the production of wine. Vine shoots have been added to wine to obtain distinctive characteristics [4] or used to obtain activated carbon [5]. Wine by-products, such as grape skin and stems, have been used to reduce the use of sulfur dioxide in the production of red wine [6]. In addition, concentrates obtained from grape skins, stems and leaves have been used in food production for their functional properties [7]. Moreover, in the olive industries, a huge variety of residues are obtained, including twigs, leaves and alperujo (olive oil pomace) [8]. Recently, Schaide et al. [9] found that the addition of olive leaf extracts during the fermentations of table olives improved its nutritional value. Olive oil pomace highlights a source of high-added value compounds such as fermentable sugars and phenols [10]. There are other relevant worldwide widespread industries such as sweet orange juice. Recent studies have characterized the orange peel as a source rich in bioactive molecules (polyphenols, carotenoids, dietary fiber and others) [11].

Thus, the agri-food residue management to its valorization is an option to change the way we produce our industrial production into a sustainable circular economy based on the reduction in the consumption of raw materials and waste food recovery [3,6]. In addition, this could be part of the strategy to reach the Sustainable Development Goals (SDG) of the United Nations, which includes food process investigation and redesign, as well as other possible solutions such as integrated supply chain models, redistribution, recovery or disposal [12].

Furthermore, according to International Oil Council (IOC) data for the 2021/2022 campaign, Spain is the leading producer of table olives, with an estimated production of table olives of 22.6% worldwide [13]. Extremadura, a southwestern Spanish region, is the second table olive producer in Spain after the Andalucía region [14]. The main industrial elaboration processes are Spanish-style, Californian-style and Natural. One of the main problems in the California-style table olive sector is that this style of production must undergo a sterilization process that causes the synthesis of acrylamide, a potentially toxic substance. The European Commission Recommendation (EU) 2019/1988 included olives stored in brine solutions in the list of foods in which acrylamide levels must be controlled [15]. Furthermore, different researchers have studied the development of this substance in olives [16–18] and have established different ways to reduce the acrylamide formation. In this sense, other authors [19–21] have established industrial strategies, such as the evaluation of the state of maturity, the application of a washing treatment before oxidation or the addition of different additives in the cans, to reduce the formation of this toxic substance. Researchers [21,22] have studied the effect of different amino acids (cysteine or proline) and the effect of calcium chloride, in the development of acrylamide in the cell wall of the table olives [17,19], and the consequence of the addition of certain natural phenolic compounds (garlic or olive leaf extract) or synthetic phenols (hydroxytyrosol or tyrosol) in the acrylamide formation in Californian-style ripe table olives [18–20]. These researchers have established that acrylamide is mainly synthesized by Maillard reaction, although in the last years, different mechanisms have appeared to be involved in the acrylamide formation associated with fat-rich foods such as table olives [18–21].

Alternatively, the aromas of the olives change when they are submitted to thermal sterilization. That could be the reason why this stuffed product is not flavored or put on the market. Therefore, it might be necessary to study these olives with different aroma concentrations to find out the optimal flavored addition to obtain a characteristic aroma in the product to be marketed. In this sense, olives filled with flavored hydrocolloids are an interesting way to obtain a new product with a characteristic aroma. The hydrocolloids, such as polysaccharides, proteins or lipids, are usually used to stuff olives with natural or artificial aromas in Spanish-style table olives. Sánchez et al. [23] studied the effect of the olfactory pattern of Californian-style black olives after being subjected to flavored hydrocolloids with the aim to reduce the cooking effect that appears after the thermal treatments [24]. New studies would be of interest to determine the effect of other by-

products and novel types of olives stuffing to control the production of acrylamide, and thus, to obtain a product with a high economic value from different residues from food industries.

This work is focused on using the by-products of different agri-food industries (wine, olive oil and orange sweet juice) to obtain extracts rich in bioactive compounds for its revalorization and to analyze the ability of these extracts to reduce the acrylamide concentration with the higher sensory characteristic. The elaboration of oxidized black olives stuffed with aromatized hydrocolloids was also studied in order to obtain a sensorially commercial product with a lower amount of acrylamide.

2. Materials and Methods

A diagram of the overall experimental design is shown in Figure 1.

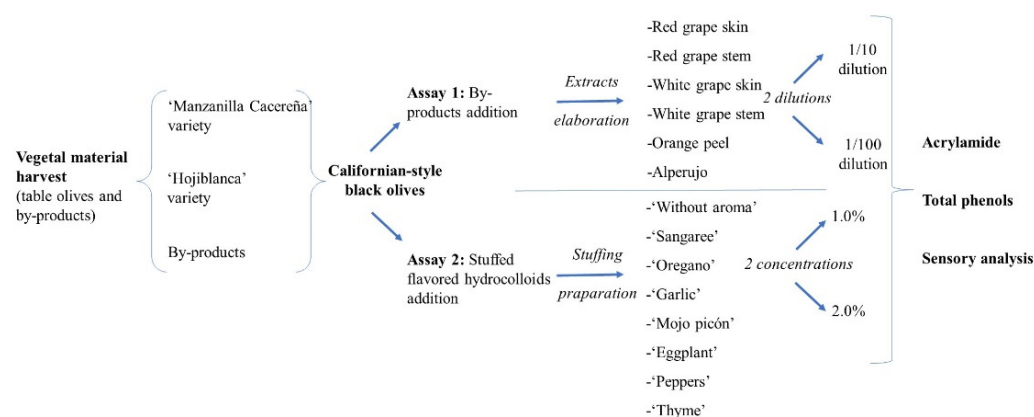


Figure 1. Diagram of the experimental design.

2.1. Vegetable material

Olives (*Olea europaea* L.) from two different varieties ('Manzanilla Cacereña' and 'Hojiblanca') at the green stage of maturation were harvested in the southwestern Mediterranean area in a test field of the Scientific and Technological Research Center of Extremadura (CI-CYTEX) (Badajoz, Spain) during the 2021/22 season. Fruits were taken from 10 trees per variety, and a total of 30 kg per variety was taken at random from the olive tree. The olives were transported to the agri-food pilot plant in ventilated storage trays and immediately stored in bins in triplicate with acetic acid (1.5%) and salt (4.0%) for 4 months. After that, olives were processed at Californian-style black olives.

At the same time, 500 g of each by-product—the skin and stem of red and white grapes, orange peel and alperujo (olive oil pomace)—were picked up from different industries of the region. Samples were vacuum-packed (Gustav Müller VS 100, Homburg, Germany) in plastic bags and after that, they were stored at frozen conditions (−80 °C).

2.2. Californian-Style Black Ripe Table Olives

Olives were submitted to Californian-style black ripe olives processes [20]. Briefly, olives were washed to eliminate the excess of acid and salt, and after that, they were submitted to a lye and air oxidation treatment until the sodium hydroxide (Sigma-Aldrich®, St. Louis, MO, United States) penetrated the olive pit. Table olives were neutralized in the same tank at pH = 7, and a ferrous gluconate solution (0.15% w/v) (Panreac, Barcelona, Spain) was applied in order to fix the black color. Olives were pitted and washed to remove the excess of additives.

2.3. Experimental Design

Two different assays were performed to evaluate the effect of phenolic extracts and stuffed flavored hydrocolloid on the acrylamide formation in Californian-style black olives.

2.3.1. Assay 1

The present assay was performed to determine the effect of the acrylamide formation after the addition of different by-products in Californian-style black olives from the 'Manzanilla Cacereña' variety. The by-products were defrosted 48 h before being processed. After that, samples were dried in a conventional oven model 210 (Selecta[®], Barcelona, Spain) for 12 min at 120 °C until samples reach a moisture loss of 20% [25]. The different types of by-products were ground with a commercial thermobeater, and the bioactive compounds were extracted at two dilutions (1:10 and 1:100) with Milli-Q water (Millipore, Bedford, MA, USA). The mix was introduced in a cooking machine at 60 °C for 1 h. The extracts were filtered (FILTER-LAB, Barcelona, Spain) and centrifuged (Thermo Scientific Sorvall Legend XT/XF centrifuge, with a F13-14x50c carbon fiber rotor, Thermo Fischer Scientific, Waltham, MA, USA) at $21,036 \times g$ at 4 °C for 10 min to remove solid particles from it. The liquid fraction was introduced in an amber-colored glass bottle (1000 mL) with 2% of sodium chloride (Panreac, Barcelona, Spain), and ferrous gluconate (0.15% *w/v*) (Panreac, Barcelona, Spain) and $2 \text{ g} \cdot \text{L}^{-1}$ calcium chloride (*w/v*) (Tetra Chemicals Europe, Helsingborg, Sweden) were added and stored at 4 °C. Californian-style black olives from the 'Manzanilla Cacereña' variety were packed (150 g) in cans with the extract's solution prepared.

2.3.2. Assay 2

The assay was performed to assess the influence of the addition of different stuffed flavored hydrocolloids in the acrylamide formation in Californian-style black olives from 'Hojiblanca' variety. After the elaboration process, olives were placed into jars (250 mL) with a solution containing 2% sodium chloride and ferrous gluconate (0.15% *w/v*). Next, the solution was removed, and olives were stuffed with the following flavored hydrocolloids: Control 1 (without hydrocolloid), Control 2 (with hydrocolloid but without aroma), 'Sangaree,' 'Oregano,' 'Garlic,' 'Mojo picón,' 'Eggplant,' 'Peppers' and 'Thyme.' The hydrocolloids [23] were first elaborated by mixing two parts of sodium alginate (Saporepuro, Torino, Italy), one part of guar gum (Saporepuro, Torino, Italy) and one part of the corresponding aroma (Neroliane, Grasse, France) at two concentrations: 1.0% (dilution 1) and 2.0% (dilution 2). The additives were mixed using a commercial mixer for 1 min with 100 mL of Milli-Q water. One batch of the table olives was also filled with an unflavored hydrocolloid. Olives were manually filled using a syringe. The stuffed olives were placed into a 7.5% calcium chloride solution for 30 s and, later, into another 0.25% calcium chloride solution for 24 h. The solution was then removed, and cans were sterilized. Five replicates per sample were prepared. Olives were introduced in cans (150 g) with a new brine solution containing sodium chloride (4% *w/v*) (Panreac, Barcelona, Spain), ferrous gluconate (0.15% *w/v*) (Panreac, Barcelona, Spain) and $2 \text{ g} \cdot \text{L}^{-1}$ CaCl_2 (*w/v*) (Tetra Chemicals Europe, Helsingborg, Sweden).

2.4. Thermal Sterilization Treatment

Californian-style black olives elaborated by the two different assays were submitted to a sterilization process using an industrial autoclave. The F0 value (accumulated lethality) was 15 min, and it was calculated monitoring the olives inside the can using a temperature sensor (TC, Uxbridge, UK) connected to a computer [20]. The F0 value was expressed as the equivalent treatment duration required to reduce the initial microbial load. After the sterilization process, cans were stored at room temperature until analysis. All experiments were performed in triplicate.

2.5. Analytical Procedures

2.5.1. Extraction and HPLC-MS-QQQ Analysis of Acrylamide

The protocol used for acrylamide analysis in table olives was described by Fernández et al. [26]. An Agilent 1290 Infinity II liquid chromatograph (Agilent Technologies, Santa Clara, CA, USA) equipped with an Agilent Jet Stream electrospray ion source operating in positive ion mode and coupled to an Agilent 6460 triple quadrupole mass spectrometer

(Agilent Technologies) was used for the acrylamide determination. The results were expressed as nanograms of acrylamide per gram of sample (ng g^{-1}).

2.5.2. Total Phenols

Total phenolic compounds were analyzed using Folin–Ciocalteu reagent as the oxidizing agent and gallic acid as standard [27]. The results are expressed as milligrams of gallic acid per 100 g of sample ($\text{mg GAE } 100 \text{ g}^{-1}$).

2.5.3. Sensory Analysis

Californian-style black olives submitted to the different assays were evaluated by a tasting panel set in the Technological Institute of Food and Agriculture (CICYTEX-INTAEX) facilities, following the recommendations for tasting room conditions and the indications established by the IOC [28]. Samples were introduced in a standard glass jar covering the bottom of the glass and arranged in a single layer at 25°C . Eight trained tasters evaluated the table olives studied by olfactory and taste perception. A final evaluation score was also provided in a scoreboard designed for the purpose of this experiment to evaluate the intensity of the positive and negative attributes according to a 10 cm structured scale. The positive attributes evaluated were 'Olive,' 'Fruity,' 'Herbal,' 'Sweet' and 'Wine' aroma, while the negatives were 'Dry odor,' 'Fermented,' 'Alcohol' 'Oily' and 'Cooked' defect. A final evaluation score was provided in both assays. The intensity and type of off-odor perceived by the tasters was assessed on a structured scale from 0 to 10 to evaluate positive and negative attributes. Sensory evaluation outcomes were expressed as average values in triplicate. Values were considered reliable when coefficient of variation (CV) < 20% [28].

2.6. Statistical Analysis

A one-way ANOVA was used followed by Tukey's multiple range test to establish statistically significant differences between the different phenol additions on the table olive varieties. Significance was set at $p < 0.05$. SPSS 18.0 software was used for statistical analysis (SPSS Inc., Chicago, IL, USA). Data are expressed as means and standard deviations (SD).

3. Results and Discussion

3.1. Assay 1: Influence of By-Products Phenolic Extracts in Acrylamide Content and Sensory Characteristics

The results of the concentration of acrylamide in sterilized table olives of the 'Manzanilla Cacereña' variety after adding different agro-industrial by-products are shown in Table 1.

The acrylamide concentration in olive without the addition of extracts was 205 ng g^{-1} . After the addition of the different extracts to the govern liquid of Californian-style black olive, the concentration of acrylamide was reduced in all the extracts compared to the control without adding by-products or hydrocolloids. The 1:10 dilution showed the greatest activity in reducing the acrylamide concentration. White skin and Orange peel extracts at a 1:10 dilution were the ones that reduced acrylamide the most, with a reduction of 30% and 32%, respectively, compared to the Control. Instead, the least efficient extract for reducing acrylamide was Alperujo, with very high acrylamide concentrations close to the value obtained for Control sample at the 1:100 dilution. The behavior of Alperujo extract was very similar to the other dilutions, with a low capacity to reduce acrylamide (12.9%).

Table 1. Acrylamide concentration (ng g^{-1}) of ‘Manzanilla Cacereña’ table olive variety after the addition of different agro-industrial by-products. Different lowercase letters mean a statistically significant difference between by-product extracts (Tukey’s test $p < 0.05$ $n = 3$). Different capital letters mean significant differences between the different dilutions (Tukey’s test $p < 0.05$ $n = 3$).

Samples	Dilution	Acrylamide
Control	—	205.2 ± 10.8 aA
Red skin	1:10 dilution	88.4 ± 7.2 c
	1:100 dilution	183.2 ± 12.1 B
Red stem	1:10	76.5 ± 9.8 d
	1:100	161.2 ± 14.4 C
White skin	1:10	71.4 ± 8.6 d
	1:100	139.1 ± 14.8 D
White stem	1:10	62.2 ± 7.4 e
	1:100	108.1 ± 11.2 F
Orange peel	1:10	65.3 ± 5.2 e
	1:100	121.6 ± 13.7 E
Alperujo	1:10	168.3 ± 13.3 b
	1:100	189.6 ± 11.2 B

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The reduction of the acrylamide values in olive after the addition of different extracts can be related with the concentration of phenolic compounds (Figure 2). Different researchers [21,29,30] have studied strategies to reduce acrylamide by the addition of exogenous additives, such as phenolic compounds with high antioxidant activity, to mitigate the synthesis of the acrylamide. The results showed that olives with Orange peel at a 1:10 dilution were the ones with the highest concentrations of phenolic compounds ($87.6 \text{ mg GAE } 100 \text{ g}^{-1}$), followed by Red skin at a 1:10 dilution ($75.6 \text{ mg GAE } 100 \text{ g}^{-1}$). Table olives with Alperujo extracts presented a low content of phenolic compounds ($19.6 \text{ GAE } 100 \text{ g}^{-1}$). There was a negative correlation between the concentration of total phenolic compounds and acrylamide. Olives with the highest total phenols had the lowest acrylamide concentration. Fernández et al. [31] indicated that acrylamide reduction mitigation strategies in Californian-style black olives with the addition of olive leaf extracts. Kotsiou et al. [32] also studied how the addition of phenolic compounds to emulsion systems reduce acrylamide formation.

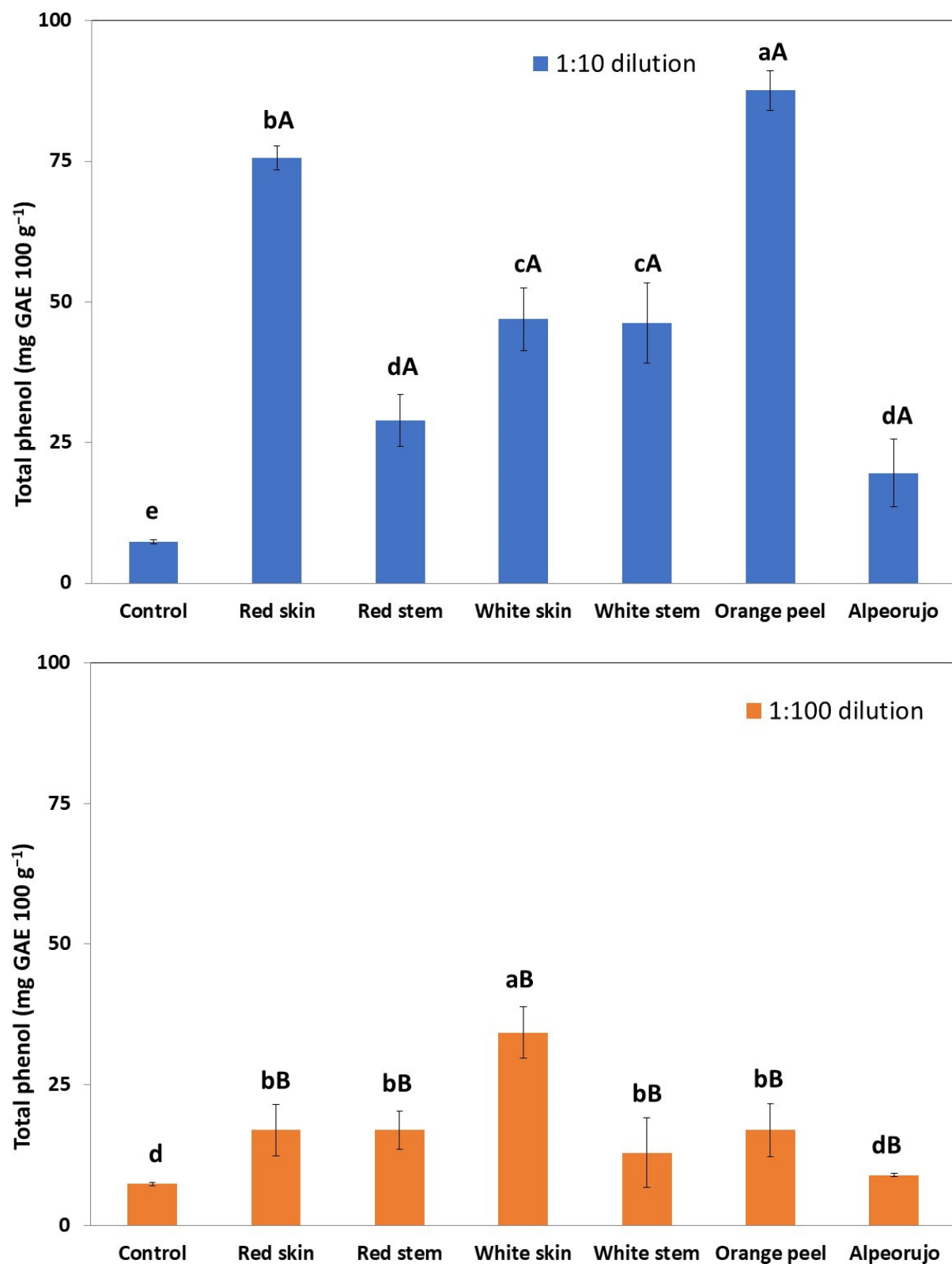


Figure 2. Total phenol (mg GAE 100 g⁻¹) of ‘Manzanilla Cacereña’ table olive variety after the addition of different agro-industrial by-products. Different lowercase letters mean a statistically significant difference between by-product extracts (Tukey’s test $p < 0.05$ $n = 3$). Different capital letters mean significant differences between the different dilutions (Tukey’s test $p < 0.05$ $n = 3$).

The seeds have a high concentration of antioxidants, so they are a type of by-product used to reduce the concentration of acrylamide [33]. In addition, other residues, such as the citrus and table olive industry and oil, are also sources of phenolic compounds [9,34]. These by-products can be used as sources of antioxidants added to different foods. In this sense, Rivas et al. [7] studied the chemical composition of skins, stems and lees from red grape vinification obtaining a higher concentration of total phenolic compounds in the stems (138.6 mg GAE 100 g⁻¹) and the skins (92.8 mg GAE 100 g⁻¹). It was observed that many natural extracts with antioxidant properties reduced acrylamide levels. In this sense, Bassama et al. [35] studied the effect of six phenolic compounds (gallic acid, ferulic acid, coumaric acid, caffeic acid, catechin and epicatechin) on the reduction of acrylamide, but did not find a mitigating effect. However, Biedermann et al. [36] found a weak reduction of acrylamide by adding ascorbic acid to potato fruits. Mildner-Szkudlarz et al. [37] studied the mitigating effect of acrylamide after adding phenolic compounds to bread in concentrations of 0.1%, managing to reduce the concentration of acrylamide (16.2–95.2%).

Other researchers [38] did not find a correlation between phenolic compounds and acrylamide in potato chips. This is because not all antioxidants have an inhibitory effect on acrylamide formation. Antioxidants that inhibit acrylamide formation may be those that react with the intermediates of the Maillard reaction, such as the flavonoids epicatechin, through an electrophilic aromatic substitution mechanism with sugar fragments of 2–4 carbon atoms [29]. Another factor influencing acrylamide formation is the position of the 3′/4′-dihydroxy groups of the hydroxyl groups present in the aromatic ring of flavonoids and in polyphenolic compounds that do not have aldehyde groups in their structure and that have high hydroxyl groups are also decisive in acrylamide inhibition [39]. Finally, there are compounds that can react with acrylamide already formed by Michael addition reactions or radical-induced elimination when these are unstable or in oxidized form, so that they can attack acrylamide [29]. For all the above, acrylamide concentration in Californian-style table olive enriched with by-product extracts gather the requirements to mitigate acrylamide thanks to bioactive molecules.

The results of sensory analysis carried out by a tasting panel with trained personnel are shown in Table 2. The positive attributes evaluated were ‘Olive,’ ‘Fruity,’ ‘Herbal,’ ‘Sweet’ and ‘Wine,’ while the negative attributes were ‘Dry odor,’ ‘Fermented,’ ‘Alcohol,’ ‘Oily’ and ‘Cooked.’ These results showed significant differences between the different aromas and dilutions. There was not significant difference for both dilutions in the by-products Red skin, Red stem, White skin and White stem in the ‘Olive’ attribute. It should also be considered that for the dilution 1:100, the score of ‘Olive’ attribute was higher than in the dilution 1:10. This could be due to the intensity of the extracts in the 1:10 dilution, being more important for the panelist in terms of flavor and odor. Alperujo was the by-product with the lowest ‘Olive’ and ‘Fruity’ attributes contribution and with the presence of ‘Oily’ smell negative attribute. The ‘Cooked’ defect was only detected in the Control due to the effect of the extracts in the samples masking the negative attribute. On the other hand, ‘Dry odor’ was detected for the Red skin and Red stem 1:10 dilution, which can be related to red wine mold or humidity attributes. The extracts with more positive attributes and not negative ones were Orange peel and White stem. The ‘Final evaluation’ for both extracts was the highest between all the samples with a score of 8.3 and 6.7 for the White stem 1:10 and 1:100 dilution, and 8.5 and 7.1 for the Orange peel 1:10 and 1:100 dilution.

Table 2. Sensory analysis of stuffed Californian-style black olives with the addition of different concentrations of by-products. Different lowercase letters mean a statistically significant difference between the dilution 1:10 (Tukey's test $p < 0.05$). Different capital letters mean significant differences between the dilution 1:100 (Tukey's test $p < 0.05$).

Samples	Dilution	Positive Attributes					Negative Attributes					Final Evaluation
		Olive	Fruity	Herbal	Sweet	Wine	Dry odor	Fermented	Alcohol	Oily	Cooked	
Control	—	6.5 ± 0.9 aA	2.1 ± 0.3 dB	—	—	—	—	—	—	—	3.4 ± 0.4	5.5 ± 0.5 cB
Red skin	1:10	2.1 ± 0.3 c	4.3 ± 0.7 b	2.2 ± 0.3 ns	2.8 ± 0.4 b	6.1 ± 0.5 b	1.2 ± 0.3 ns	1.1 ± 0.1 ns	1.2 ± 0.2 b	—	—	6.0 ± 0.8 c
	1:100	3.2 ± 0.4 C	2.3 ± 0.4 B	—	1.5 ± 0.4 A	3.4 ± 0.6 NS	—	0.7 ± 0.1 b	1.0 ± 0.1 NS	—	—	5.2 ± 0.4 B
Red stem	1:10	2.3 ± 0.2 c	3.4 ± 0.4 c	2.2 ± 0.2 ns	—	4.3 ± 0.6 a	1.0 ± 0.4 ns	—	—	—	—	7.0 ± 0.6 b
	1:100	3.5 ± 0.3 C	2.5 ± 0.6 B	1.1 ± 0.1 B	—	4.0 ± 0.4 NS	—	—	—	—	—	4.8 ± 0.5 B
White skin	1:10	2.5 ± 0.4 c	3.1 ± 0.2 c	2.4 ± 0.4 ns	5.3 ± 0.7 a	—	—	1.5 ± 0.1 ns	2.5 ± 0.3 a	—	—	6.7 ± 0.9 b
	1:100	3.9 ± 0.2 C	2.1 ± 0.3 B	—	4.0 ± 0.6 A	—	—	1.2 ± 0.2 A	1.4 ± 0.4 NS	—	—	5.4 ± 0.6 B
White stem	1:10	2.4 ± 0.1 c	7.2 ± 0.8 a	3.2 ± 0.3 ns	—	—	—	—	—	—	—	8.3 ± 0.7 a
	1:100	3.6 ± 0.3 C	3.1 ± 0.5 B	2.3 ± 0.6 A	—	—	—	—	—	—	—	6.7 ± 0.5 A
Orange peel	1:10	1.7 ± 0.2 d	7.2 ± 0.6 a	—	—	—	—	—	—	—	—	8.5 ± 0.6 a
	1:100	2.3 ± 0.5 D	5.3 ± 0.7 A	—	—	—	—	—	—	—	—	7.1 ± 0.7 A
Alperujo	1:10	4.1 ± 0.5 b	2.1 ± 0.2 d	—	—	—	—	—	—	3.2 ± 0.3	—	3.1 ± 0.5 d
	1:100	5.8 ± 0.8 B	1.5 ± 0.3 C	—	—	—	—	—	—	2.4 ± 0.2	—	3.3 ± 0.3 C

3.2. Assay 2: Effect of Healthy Stuffing in Acrylamide Concentration and Consumers Acceptance

After the execution of assay 2, the concentration of acrylamide in olives filled with phenol-rich aromatic hydrocolloids was studied (Table 3).

Table 3. Acrylamide concentration (ng g^{−1}) of the ‘Hojiblanca’ table olive variety stuffed with different aromatic hydrocolloids. Different lowercase letters mean a statistically significant difference between stuffed aromas (Tukey's test $p < 0.05$ n = 3). Different capital letters mean significant differences between the different dilutions (Tukey's test $p < 0.05$ n = 3).

Samples	Dilution	Dilution
Control 1	—	332.1 ± 11.3 bB
Control 2	—	365.2 ± 9.9 aA
‘Sangaree’	Dilution 1	308.2 ± 15.4 c
	Dilution 2	195.7 ± 12.3 dC
‘Oregano’	1	240.7 ± 13.2 d
	2	120.7 ± 9.3 G
‘Garlic’	1	165.5 ± 12.1 f
	2	120.5 ± 8.2 G
‘Mojo picón’	1	195.5 ± 11.8 e
	2	135.6 ± 12.4 F
‘Eggplant’	1	180.6 ± 9.9 e
	2	150.1 ± 14.4 E
‘Peppers’	1	240.5 ± 14.3 d
	2	180.1 ± 12.4 D
‘Thyme’	1	150.5 ± 12.6 g
	2	90.7 ± 9.6 H

It can be observed that acrylamide was detected in the samples after heat treatment and subsequent stuffing. The levels of this compound ranged between 332.1 ng g^{-1} and 365.2 ng g^{-1} for the proposed controls: olives stuffed without and with hydrocolloids (1 and 2). Olives stuffed with hydrocolloid (Control 2) presented a higher concentration of acrylamide, although both controls presented significant differences. This may be due to the composition of the hydrocolloid [23] which, when subjected to high temperatures, leads to the degradation of the stuffing. The carbohydrate part of the hydrocolloid has contributed in the Maillard reaction to synthesize acrylamide [18,40]. It can be concluded that acrylamide was already present in the control samples before adding any other additive to the olive.

In the case of the aromatic fillers, the values of acrylamide concentration were comprehended between 165.5 ng g^{-1} and 308.2 ng g^{-1} for dilution 1 and between 195.7 ng g^{-1} and 90.7 ng g^{-1} for dilution 2, with significantly different values for both. The difference between the ranges of concentrations is coherent with the dilutions carried out. For the dilution 2, 45% less acrylamide was found due to the inhibition activity of the bioactive stuffing. The set of studies that support the appearance of this toxic compound after sterilization are well known in the literature [16,21]. Similarly, there has been research demonstrating the use of olive industry by-products as healthful additives [30,41]. That said, we are not aware of any studies that have proposed the use of aromatic hydrocolloid fillers as healthful by-products to improve the bioactive and organoleptic quality of California-style black table olives. Delving further into the behavior of the aromatic fillers, it can be observed that for the 'Sangaree' filler, the highest acrylamide values were obtained with 308.2 ng g^{-1} and 195.7 ng g^{-1} for dilutions 1 and 2, respectively. The filler that mitigated the most the final acrylamide concentration was 'Thyme,' with 150.5 ng g^{-1} and 90.7 ng g^{-1} , presenting between 59% and 75% less acrylamide compared to Control 2. This may be due to the richness in bioactive compounds of these spices and herbal odors, such as thyme, rosemary, green tea extracts or oregano, studied by other researchers [21,42]. In addition, it was also observed that, for dilution 1, there were no significant differences for the fillings of 'Oregano' and 'Peppers' as well as for 'Garlic' and 'Thyme' and, in the same way, for 'Mojo Picón' and 'Eggplant.' In parallel, there was no statistical difference for 'Oregano' and 'Garlic' and for 'Mojo Picón' and 'Eggplant' for dilution 2 (Table 3). In other studies, some authors have studied the application of these fillers for commercial purposes to alleviate defects in table olives produced during fermentation and industrial processing [23,43,44].

Everything discussed in the previous paragraph concords with the data for total phenols shown in Figure 3.

In the same way as for the acrylamide concentrations, the values of total phenols were significantly different from each other. In addition, Controls 1 and 2 showed the lowest levels of total phenols, with $24.5 \text{ mg GAE } 100 \text{ g}^{-1}$ and $20.5 \text{ mg GAE } 100 \text{ g}^{-1}$, respectively. As for the rest of the fillers, the aromatic 'Thyme' continued to be the one with the best bioactive capacity with $65.3 \text{ mg GAE } 100 \text{ g}^{-1}$ and $86.5 \text{ mg GAE } 100 \text{ g}^{-1}$ for dilutions 1 and 2, respectively. Recent research has found similar results for the antioxidant capacity of these hydrocolloid fillers [43,45]. As for the contaminant concentrations, there were no significant differences for the fillers 'Oregano' and 'Pimientos,' 'Garlic' and 'Thyme,' and for 'Mojo Picón' and 'Eggplant' at dilution 1. Similarly, there were no statistical differences for 'Sangaree' and 'Pimientos,' and for 'Garlic,' 'Mojo Picón,' 'Oregano' and 'Eggplant' for dilution 2 (Figure 3). The values observed after experimentation are consistent with the relationship between antioxidant compounds such as phenolic compounds and acrylamide levels. Previous research by the group has shown the inverse relationship between these two types of compounds. The higher the concentration of phenolic compounds, the greater the attenuation that occurs in acrylamide formation after high-intensity heat treatments [18,45]. Therefore, the value of using a healthy filling in the preparation of California-style ripe black olives can be appreciated.

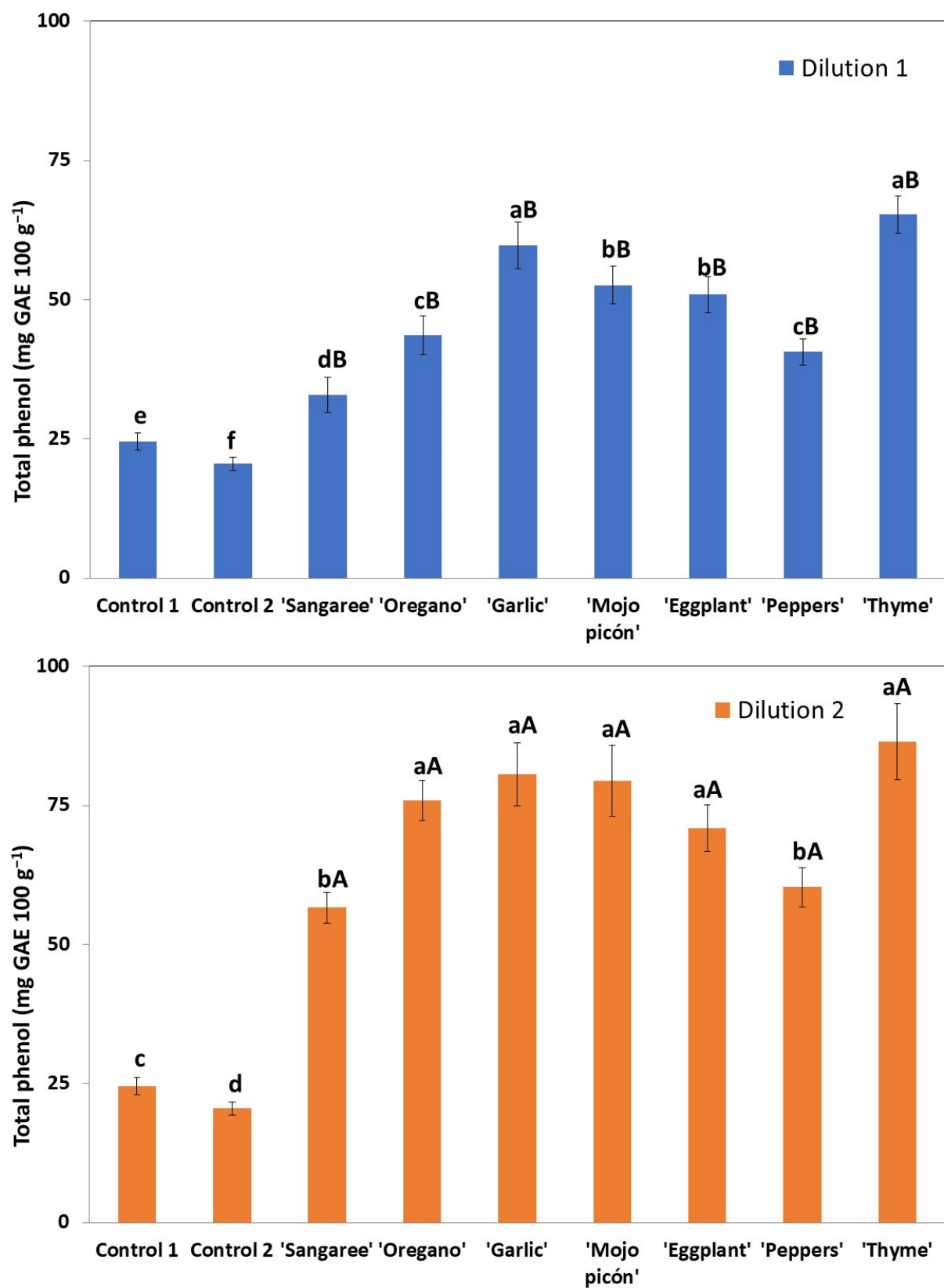


Figure 3. Total phenol (mg GAE 100 g⁻¹) of 'Hojiblanca' table olive variety stuffed with different aromatic hydrocolloids. Different lowercase letters mean a statistically significant difference between stuffed aromas (Tukey's test $p < 0.05$ $n = 3$). Different capital letters mean significant differences between the different dilutions (Tukey's test $p < 0.05$ $n = 3$).

As for the organoleptic characteristics of the samples, the sensory analysis performed for dilutions 1 and 2 is shown in Table 4.

Table 4. Sensory analysis of stuffed Californian-style black olives with the addition of different concentrations of hydrocolloids. Different lowercase letters mean a statistically significant difference between dilution 1 stuffed aromas (Tukey's test $p < 0.05$). Different capital letters mean significant differences between the dilution 2 stuffed aromas (Tukey's test $p < 0.05$).

Samples	Dilution	Positive Attributes					Negative Attributes					Final Evaluation
		Olive	Fruity	Herbal	Sweet	Wine	Dry odor	Fermented	Alcohol	Oily	Cooked	
Control 1	—	6.3 ± 0.7 aA	2.1 ± 0.2 dD	—	—	—	—	—	—	—	3.1 ± 0.3 ns	5.4 ± 0.5 d
Control 2	—	6.1 ± 0.6 aA	2.3 ± 0.3 dD	—	—	—	—	—	—	—	3.3 ± 0.2 ns	5.5 ± 0.4 B
'Sangaree'	1	2.9 ± 0.3 b	3.5 ± 0.5 c	—	—	2.1 ± 0.2	1.0 ± 0.4 ns	1.6 ± 0.2	1.2 ± 0.4 b	—	—	4.2 ± 0.2 e
	2	1.6 ± 0.4 C	4.2 ± 0.4 C	—	—	3.4 ± 0.5	2.5 ± 0.3 ns	2.5 ± 0.3	1.6 ± 0.5 b	—	—	2.1 ± 0.3 D
'Oregano'	1	2.5 ± 0.6 bc	3.9 ± 0.4 c	2.1 ± 0.3 b	1.4 ± 0.2 a	—	1.5 ± 0.3 ns	—	—	1.3 ± 0.2 a	—	6.3 ± 0.4 c
	2	1.7 ± 0.3 C	4.7 ± 0.2 C	2.6 ± 0.3 B	1.6 ± 0.2 A	—	2.4 ± 0.4 ns	—	—	1.7 ± 0.4 a	—	4.2 ± 0.5 C
'Garlic'	1	2.0 ± 0.1 c	7.3 ± 0.9 a	—	2.2 ± 0.2 b	—	—	—	—	—	—	7.5 ± 0.6 b
	2	1.2 ± 0.4 D	8.5 ± 0.8 A	—	2.8 ± 0.4 B	—	—	—	—	—	—	8.3 ± 0.6 A
'Mojo picón'	1	1.8 ± 0.3 c	7.3 ± 0.6 a	—	1.5 ± 0.1 a	—	—	—	—	—	—	8.5 ± 0.6 a
	2	1.2 ± 0.3 D	8.4 ± 0.3 A	—	2.5 ± 0.2 B	—	—	—	—	—	—	9.2 ± 0.6 A
'Eggplant'	1	2.2 ± 0.2 c	7.5 ± 0.8 a	3.6 ± 0.5 a	—	—	—	—	—	—	—	8.4 ± 0.5 a
	2	1.4 ± 0.4 D	8.9 ± 0.6 A	4.2 ± 0.8 A	—	—	—	—	—	—	—	9.3 ± 0.5 A
'Peppers'	1	2.9 ± 0.4 b	3.3 ± 0.3 c	—	—	—	—	—	—	—	—	6.4 ± 0.5 c
	2	1.7 ± 0.4 C	4.4 ± 0.5 C	—	—	—	—	—	—	—	—	5.1 ± 0.7 B
'Thyme'	1	3.1 ± 0.3 b	6.2 ± 0.7 b	3.5 ± 0.4 a	—	—	1.5 ± 0.3 ns	—	2.1 ± 0.2 a	2.3 ± 0.3 b	3.2 ± 0.3 ns	3.5 ± 0.4 f
	2	2.7 ± 0.5 B	7.4 ± 0.4 B	4.7 ± 0.4 A	—	—	2.3 ± 0.2 ns	—	2.9 ± 0.1 a	3.2 ± 0.2 b	2.4 ± 0.2 ns	2.0 ± 0.2 D

For the panelists, Control 1 and 2 were rated as statistically equal in attributes such as 'Olive' and 'Fruity,' but their defect of 'Cooked' was considered statistically non-significant. Significant differences were observed for all fillers in almost all attributes, with the exceptions of the 'Dry Odor' and 'Cooked' defect. In the case of the 'Thyme' filler, it was found that for both positive and negative attributes presented by the sample, dilution 1 and 2 were statistically equal. With respect to the positive attributes, all stuffed olives presented the attributes of 'Olive' and 'Fruity,' with 'Herbs' being the next most common and, finally, 'Wine' only presented in the 'Sangaree' stuffing. As for the negative attributes, 'Sangaree' and 'Thyme' were the fillings with the most attributes according to the tasters, although all at levels below 3.2. For all these reasons, the 'Mojo Picón,' 'Eggplant' and 'Garlic' fillings were the most appetizing to the tasters and, consequently, the ones that received the highest overall rating. The best rated filling was 'Mojo Picón' with 8.5 and 9.2 for dilution 1 and 2 respectively. The use of healthy fillers has been previously proposed together with the use of by-products accepted by consumers, looking for a sensory profile absent of bitterness and with adherence in the human diet [9,23,43].

4. Conclusions

Nowadays, the use of different industrial by-products should be an emerging initiative in food market for different reasons. This could help move the agroindustry toward a circular economy by providing added value to these by-products in order to improve

table olives quality. For this reason, bioactive extract elaboration and its addition to Californian-style black olives could be a good application of this focus. As the results show, bioactive compounds play an important role in adding extra value to the market product. The revaluation of by-products is a necessary consequence of an innovative, sustainable and reliable mindset. In addition, different presentation formats should also improve the organoleptic and bioactive aspects of table olives. It can be concluded that the concentration of by-product or hydrocolloid aroma stuffed in table olives influences the acrylamide content. White skin and Orange peel extracts at a 1:10 dilution were the most interesting in terms of acrylamide reduction, with Orange peel having the highest positive sensory attributes. The results show that the reduction of the acrylamide values in olive after the addition of different extracts can be related with the concentration of phenolic compounds. As for the hydrocolloids, a higher number of hydrocolloids aromas reduced the acrylamide content. The filler that mitigated the most the final acrylamide concentration was 'Thyme,' which presented the best bioactive capacity. One of the drawbacks of this aroma is the negative aromas sensorially detected. The use of healthy aromatic filling is an excellent strategy for that, mitigating acrylamide formation with its inclusion in the product as well as providing new ways of consuming Californian-style black olives that could be appealing to the consumer.

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