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Impact of thermal sterilization on the physicochemical-sensory characteristics of Californian-style black olives and its assessment using an electronic tongue

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ABSTRACT

The effect of thermal sterilization processes on the physicochemical parameters and sensory characteristics of Californian-style black olives, from 'Hojiblanca' and 'Manzanilla Cacereña' brine solutions, were evaluated. Two-way ANOVA showed that 'Hojiblanca' olives had a lower phenols content and defect intensity and that increasing the sterilization period resulted in a decrease in total phenols and an increase of the cooked defect. The impact of thermal sterilization was further evaluated using a potentiometric electronic tongue, which was capable of discriminating the different sterilization treatments (repeated *K*-fold cross-validation sensitivity: 89.0 ± 15.0% to 97.0 ± 6.0%). Moreover, multiple regressions allowed the prediction of phenols ($R^2 \geq 0.95 \pm 0.03$) and the intensity of the defect ($R^2 \geq 0.95 \pm 0.04$). These results point out the feasibility of the electronic tongue as an analytical tool for monitoring the effects of thermal sterilization treatments. Furthermore, the satisfactory results obtained for the brine solutions may foresee a practical non-destructive method for indirect quality assessment of table olives.

1. Introduction

Nowadays, Spain is the biggest producer of table olives, followed by Egypt, Turkey, Algeria, Greece, Syria and Morocco. According to the International Olive Council (IOC), the average worldwide production of the last five campaigns was 2,751,500 tons (IOC, 2019). Spanish production represents approximately 71% of EU production and 22% of world production (IOC, 2019). One of the most common ways of preparing table olives is Californian-style black olives. In this kind of process, olives are produced by subjecting them to several lye treatments in order to eliminate the bitterness and to obtain olives with a black color through forced oxidation. The final product is not microbiologically stable; therefore, thermal sterilization is mandatory, being usually carried out at 121–126 °C (Sánchez, García, & Rejano, 2006).

This sterilization process contributes to the formation of the flavor characteristics of the finished product, which largely depend on the olives variety used and the intensity of the thermal treatment applied (Charoenprasert & Mitchell, 2014; Pérez-Nevado, Cabrera-Bañegil, Repilado, Martillanes, & Martín-Vertedor, 2018; Tang et al., 2016). However, heat sterilization can be responsible for thermal degradation of the nutritional constituents of foods, such as antioxidant compounds (Pérez-Nevado et al., 2018) and vitamins (Ariahu, Abashi, & Chinma, 2011; Ummarino et al., 2017), by promoting their oxidative degradation. Water-soluble vitamins are often used as indices of thermal damage in vegetable materials. In fact, it is well known that ascorbic acid is heat-sensitive (Ariahu et al., 2011) while B3 vitamins (Ummarino et al., 2017) are the most stable water-soluble vitamins, being less prone to heat and/or oxygen degradation (Okmen & Bayindirli, 1999).

At industrial level, different thermal treatments are implemented for the sterilization of olive cans. In the past, the Spanish legal regulation imposed the application of a minimum amount of heat during sterilization of table olives to ensure microbiological safety, setting a

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sterilization value (or F_0 value) greater than or equal to 15 min (Royal Decree 1230/2001). Nevertheless, the application of such a severe sterilization treatment was not consensual; recently, a new Spanish legal regulation was published in which the minimum F_0 value was removed (Royal Decree 679/2016). Indeed, a recent study showed that Spanish and Portuguese commercial canned black table olives, as well as their brine solutions, suffer wide degradation of phenolic compounds due to the thermal sterilization treatments applied (Pérez-Nevado et al., 2018). Moreover, the sterilization treatments also play a key role at the sensory level, contributing to changes in the intensity of positive sensory attributes and to the appearance of possible negative attributes (e.g., cooked defect) (Casado, Sánchez, Rejano, & Montaño, 2007). Therefore, monitoring the impact of the usual industrial thermal sterilization treatments on the physicochemical and sensory characteristics of canned table olives is of the utmost relevance for both producers and consumers. Conventional analytical techniques (e.g., chromatography, UV–Vis spectrophotometry, etc.) and trained sensory panels are commonly applied to evaluate the chemical and sensory properties of olives (Lanza & Amoruso, 2020; Lodolini et al., 2019; López-López, Sánchez-Gómez, Montaño, Cortés-Delgado, & Garrido-Fernández, 2019; Martín-Vertedor et al., 2020; Marx et al., 2017c; Mastralexi, Mantzouridou, & Tsimidou, 2019; Moreno-González, Juan, & Planas, 2020; Pérez-Nevado et al., 2018; Sánchez, López-López, Cortés-Delgado, de Castro, & Montaño, 2020). However, as recently pointed out by Lanza and Amoruso (2020), evaluation of the sensory attribute of table olives by sensory panels (composed of 8–10 expert/selected tasters and a panel leader) is a difficult task, it being highlighted that some sensations (e.g., hardness) are more easily perceived than other attributes (e.g., saltiness, bitterness, acidic, fibrousness and crunchiness).

In recent years, electrochemical sensors, such as the electronic tongue (E-tongue), have emerged as powerful sensory tools (i.e., taste sensor devices) that allow the qualitative and quantitative assessment of the positive and negative gustatory sensations of table olives (Marx et al., 2017b, 2017c, 2017a; Rodrigues et al., 2019). The literature also reports the use of potentiometric and voltammetric E-tongues coupled with different chemometric tools for the physicochemical and sensory analysis of olive oil (Apetrei & Apetrei, 2013; Apetrei, Ghasemi-Varnamkhasti, & Apetrei, 2016; Rodrigues, Dias, Veloso, Pereira, & Peres, 2016). E-tongues comprising lipid-polymeric sensor membranes can, theoretically, be used to assess chemical and sensory properties since electrostatic or hydrophobic interactions can be established between the lipid membranes and polar compounds of a target sample (Kobayashi et al., 2010; Marx et al., 2017a, 2017b, 2017c). Lipid-polymeric membranes contain polar (negative and/or positive polarities) and non-polar regions, allowing the establishment of the above-mentioned interactions and promoting the potentiometric assessment of samples' polar compounds some of them related to the sensory sensations of a specific sample. In fact, it has been reported that lipid-polymeric membranes comprising different combinations of lipid additives (e.g., methyltrioctylammonium chloride, oleic acid, oleyl alcohol, and octadecylamine) and plasticizers (e.g., bis(1-butylpentyl) adipate, dibutyl sebacate, dioctyl phenylphosphonate, 2-nitrophenyl octyl ether and tris(2-ethylhexyl)phosphate) show a quantitative potentiometric response to different chemical compounds including phenolic aldehydes, esters, alcohols, aldehydes (Slim et al., 2017), acids, salts, caffeine and quinine (Marx et al., 2017a; 2017b). E-tongues have been recognized as simple, green and user-friendly techniques, with low analytical cost and short analysis time, allowing the analysis of a large number of samples per day. However, to date, the use of potentiometric E-tongues for evaluating the physicochemical and sensory attributes of table olives has required analysis of the olive transformed into an aqueous paste (Rodrigues et al., 2019), therefore being a destructive technique. Besides that, in some situations, simultaneous analysis of the brine solutions is also required, increasing the complexity and the duration of each analysis (Marx et al., 2017a; 2017b; 2017c). Thus, the possibility of using this emerging analytical tool as a non-destructive technique would be of utmost relevance.

Thus, the purpose of this study was to evaluate Californian-style black olives submitted to different thermal treatments using an E-tongue, aiming to discriminate the thermal treatments applied and to quantify the intensity of the sensory sensations perceived by a trained sensory panel. This study aims to give a contribution for settling E-tongues as useful and fast non-destructive analytical tools for the sensory evaluation of table olives that can be implemented at industrial level, in compliance with the recommendations of the European Union for quality improvement of the final elaborated product. For that, the possibility of enhancing the E-tongue signal sensitivity, by incorporating sensor lipid membranes with a larger contact surface and greater thickness, is investigated.

2. Material and methods

2.1. Samples

Olives (*Olea europaea* L.) from two cultivars were obtained from olive producers that collaborate with the Researcher Center "La Orden-CICYTEX" (Badajoz, Spain). The olive groves were located in the "Vegas Bajas del Guadiana" region and olives were collected during the crop season 2018/19. 'Hojiblanca' and 'Manzanilla Cacereña' olive varieties were harvested at green maturation stage with the ripeness index 1 (green) according to the color of the skin and flesh evaluation as proposed by Uceda and Frías (1975). After harvest, olives were stored in industrial tanks of 16,000 L of capacity. Olives were stored in triplicate in a table olive company set in the northwest of Extremadura (Spain). Table olives processing were done according to the Californian-style black olive (Martín-Vertedor et al., 2020). Briefly, olives were treated with NaOH to remove the bitter taste, being air bubbled continuously to promote the oxidation of the olives. After neutralisation with lactic acid and CO_2, a ferrous gluconate solution (0.15%, w/v) was applied to fix the color. Olives were then washed to remove any ferrous gluconate residual. After the stone removal, olives were packed (150 g) in cans with a solution containing 2% of NaCl and 0.015% (10–40 ppm) of ferrous gluconate.

2.2. Thermal sterilization experimental treatments

Ferrous gluconate (0.01%) and salt (2%) were added to cans that contain 150 g of olives and its respective brine. Cans were sterilized at 121 ± 3 °C appling different thermal treatments combinations in order to give a reduction ratio in microorganisms taking into account their cumulative lethality ("F_0 value"). Two Pt100 temperature sensors (TC, United Kingdom), were stuck, each one on an olive and placed in the center of two cylindrical cans (diameter: 6.7 cm; height: 10.2 cm). The sensors were connected to a computer, allowing to record the temperature profile inside the sealed cans during the processing time, ensuring that the desired heat penetration was attained. The F_0 value was calculated using the recorded heat penetration data (by integrating the time-temperature profile recorded by each sensor and for each treatment) and expressed as the equivalent treatment duration (in minutes) required to reduce the initial microbial load, at a specified reference temperature, to a desired value, assuring the inactivation or the reduction of thermo-resistant spoilage bacterial spores (Tang et al., 2016). Thus, table olives from both olive cultivars were submitted to four different thermal sterilization treatments corresponding to four different F_0 values, namely, F_0 = 10 min (T1), F_0 = 15 min (T2), F_0 = 20 min (T3) and F_0 = 25 min (T4). All experiments were done in triplicate and the results based on the experimental data were expressed as mean values ± standard deviation. During storage, cans were protected from light exposure and kept at room temperature until analysis.

2.3. Analyses

2.3.1. Physicochemical parameters

pH and total chlorides contents were assessed after the elaboration process. 10 g of olives samples were added with distilled water (25 mL) and mixed with an T-18 Basis Ultra-Turrax® Homogenizer equipment (IKA, Germany). The filtrated solution was filled up to 100 mL with distilled water to perform pH and chlorides. For brine, 100 mL of the solution was directly used to make the analyses. The pH was evaluated on a pH meter (Crisol, Model Basic20) and total chlorides were analyzed by Mohr's method (Schaide, Cabrera-Bañegil, Pérez-Nevado, Esperilla, & Martín-Vertedor, 2019). The results were expressed as relative percentages (%). The samples were analyzed in triplicate.

2.3.2. Total phenols

Total phenols (TP) were analyzed using Folin–Ciocalteu method (Savini et al., 2017; Schaide et al., 2019) based on colorimetric oxidation/reduction reactions, using gallic acid as the standard and the Folin–Ciocalteu reagent as the oxidising agent. 1 mL of the diluted extract was mixed with 1 mL of Folin–Ciocalteu reagent (Panreac, Barcelona, Spain), 1 mL of saturated Na_2CO_3 and 7 mL of distilled water. The solution was agitated in a vortex (VWR® Vortex Mixers, 230 V) during 1 min after each reagent addition. After 90 min of incubation, the spectrophotometric absorbance was measured at 725 nm against a blank solution, using a UV–Vis Spectrophotometer (GENESYS™ 10, from Thermo Scientific™). A calibration curve (dynamic concentration range: 0.001–1 mol/L, $R^2 = 0.9997$) was constructed using Gallic acid (purity ≥ 99%, from ExtraSynthese). The results were expressed as grams of gallic acid per 100 g of sample (g GAE 100 g^{-1}).

2.3.3. Sensory analysis

The sensory analysis of the table olives was perfomed by a trained panel of eight panelists, of both sexes and different ages, belonging to the multidisciplinary team of the CICYTEX research center (Extremadura, Spain). The sensory panel was trained followed the IOC's regulation (IOC, 2011). For the sensory analysis of the olives, a score board was prepared and the sensory properties of olive fruits, including color, bitter, salty, acid and defects (off-flavors) were evaluated by the panelists according to the IOC standardized norm (IOC, 2011). The sensory defects were also evaluated in brine solution.

2.3.4. Electronic tongue analysis

An E-tongue was applied to discriminate Californian-style black olives of 'Hojiblanca' or 'Manzanilla Cacereña' according to the applied thermal sterilization treatment (T1: $F_0 = 10$ min; T2: $F_0 = 15$ min, T3: $F_0 = 20$ min, and T4: $F_0 = 25$ min; all at 121 ± 3 °C).

2.3.4.1. E-tongue device. The lab-made E-tongue (non-commercial device) included two cylindrical sensor arrays. As previously described (Marx et al., 2017a), each array had 20 lipid polymeric cross-sensitive membranes containing 3% of a lipid additive, 32% of a plasticizer and 65% of polyvinyl chloride, aiming to increase the sensors' sensitivity towards the chemical compounds responsible for both chemical and sensory properties of the table olives, the E-tongue device comprised thicker lipid membranes (~2 mm) with higher superficial contact surface (~20 mm^2) compared to those previously used by Marx et al. (2017a, 2017b, 2017c). A multiplexer Agilent Data Acquisition Switch Unit (model 34970 A) and an Agilent BenchLink Data Logger software were used for registering the membranes signal profiles, being the 40 potentiometric signals recorded during 5 min. An Ag/AgCl double-junction glass electrode (Crison, 5241) was used as the reference electrode. The E-tongue was stored at room temperature and immersed in a HCl cleaning solution (0.01 mol/L). As in previous studies, the sensor codes included the letter S for sensor identification, the array number (1: or 2) and the membrane number (1–20) (Marx et al., 2017a).

2.3.4.2. Sample preparation and potentiometric analysis. Representative potentiometric profiles of the table olives ('Hojiblanca' and 'Manzanilla Cacereña'), produced according to the Californian-style black olive process, were gathered by the lab-made E-tongue (table olives from 6 cans (replicas) were evaluated for each of the 4 thermal treatments and for each olive cultivar, totalizing 48 samples). For analysis, a fine grained aqueous olive paste was obtained using IKA T-18 Basic Ultra Turrax® Homogenizer equipment (using 25 g of olives without stone plus 75 mL of deionized water).

The brine solutions were also analyzed with the E-tongue (brine samples from 6 cans (replicas) were evaluated for each of the 4 thermal treatments and for each olive cultivar, totalizing 48 samples). To keep a similarity to the olive assay, the brine solution was diluted using deionized water (25 mL of brine and 75 mL of deionized water, ensuring a similar proportion of that used for obtaining the olive aqueous paste).

All assays were performed at approximately 20 °C under agitation (MSL-8 magnetic stirrer, VELP® Scientifica, Italy). The E-tongue was immersed in each simple being the 40 sensors signals recorded after a 5-min stabilization period, which was monitored at each 10-s interval (Marx et al., 2017a). To minimize the risk of signal drift the assays were carried in the same day ensuring that a negligible signal variation occurred as pointed out by Prata et al. (2018) and Veloso, Dias, Rodrigues, Pereira, and Peres (2016). This issue was also experimentally confirmed by repeating the analysis for the same HCl standard solution (0.01 mol/L) during the analysis day.

2.4. Statistical analysis

A two-way ANOVA with interaction was used to evaluate the effect of olive cultivar and of the thermal sterilization treatment on the quality composition of Californian-style black olives and the respective brine solutions. Depending on the statistical significance of the interaction effect and on the type of interaction found (additive or non-additive/disordinal effect) the Tukey's multiple range test or the estimated marginal mean plots were further used to discuss at which level the factors influenced the dependent variables under study (Field, 2009). Statistical significance was assumed at a *P*-value level lower than 0.05. ANOVA was performed using the SPSS 18.0 software (SPSS Inc., Chicago, IL, USA).

The discrimination power of the E-tongue to identify the different thermal treatments applied during the table olives sterilization was evaluated, for the olive aqueous pastes and the brine solutions, using a linear discriminant analysis (LDA) coupled with the meta-heuristic simulated annealing (SA) variable selection algorithm (Bertsimas & Tsitsiklis, 1993; Cadima, Cerdeira, & Minhoto, 2004; Kirkpatrick, Gelatt, & Vecchi, 1983). The SA meta-heuristic algorithm was used to identify the best sub-set containing the lower number of non-redundant E-tongue sensors (Rodrigues et al., 2016; Veloso et al., 2016). The LDA performance was checked using two internal cross-validation (CV) variants, namely the leave-one-out CV (LOO-CV) and the repeated *K*-fold-CV (*K* equal to 4 folds, allowing keeping 25% of the initial data for validation purposes, being the data randomly split for 10 ×). The former is considered an over-optimistic internal validation technique and the latter aims to overcome this possible limitation and to minimize overfitting risks. Variable scaling and centering procedures were implemented as data normalization procedures. The LDA models outputs were graphically evaluated using 2D plots of the two most significant linear discriminant (LD) functions and by calculating the sensitivity values (i.e., the percentage of samples correctly classified into the pre-established groups).

Multiple linear regression models (MLRM) together with the SA algorithm were established based on E-tongue data, and used to determine the contents of different chemical and sensory attributes of the table olives as well of their brine solutions experimentally assessed (UV/Vis spectrophotometry, liquid chromatography and trained sensory panel). The quality of the E-tongue-MLR-SA models was evaluated using the coefficient of determination (R^2) and the root-mean-square error (RMSE) for the LOO-CV and the repeated K-fold-CV procedures. Finally, the accuracy of the E-tongue-MLR-SA models was compared against the analytical conventional techniques used for assessing the chemical and sensory data (Roig & Thomas, 2003a; b). The E-tongue-MLR-SA models were developed using the potentiometric profiles recorded by the 40 E-tongue sensors (−0.13 V to +0.36 V and −0.08 V to +0.35 V for aqueous olive pastes and diluted brine solutions, respectively) during the analysis of the aqueous olive pastes (6 independent olive bottled samples per heat treatment) or the respective diluted brine solutions. The SA algorithm was applied for identifying the non-redundant potentiometric data.

The LDA and MLRM were performed using the Sub select (Cadima et al., 2004) and MASS (Venables & Ripley, 2002) packages of the open source statistical program R (version 2.15.1), at a 5% significance level.

3. Results and discussion

3.1. Physicochemical and sensory properties of table olives and brine solution

The physicochemical parameters and sensory properties of 'Manzanilla Cacereña' and 'Hojiblanca' olives, after being processed as Californian-style black olives and submitted to thermal treatments (T1 - T4), were evaluated. The results from the two-way ANOVA with interaction (Table 1) showed that the color, TP content, salty sensation intensity and cooked defect intensity of olives as well as the TP contents and cooked defect intensity of the respective brine solutions were significantly influenced by the olive cultivar and/or the thermal sterilization treatment. For each of the mentioned parameters, the interaction effect (cultivar × thermal treatment) was not statistically significant or if a significant statistical effect was found (P-value < 0.05) an additive behaviour (i.e., non-disordinal effect) was observed (Fig. 1), since no crossing lines were observed. This fact allowed discussion of the individual effect of each main factor. Concerning the other physicochemical and sensory attributes studied (pH and total chlorides of both olives and brine solutions as well as bitter and acid sensations of olives), no statistically significant effect or interaction effect was found (P-value > 0.05). This was expected since usually these parameters are standardized by table olives producers to typify the product, aiming to meet consumers' expectations.

The results (Table 1 and Fig. 1) showed that, regardless of the intensity of the thermal treatment, 'Manzanilla Cacereña' table olives had a significantly lower color intensity than 'Hojiblanca' olives, despite both of them being produced as Californian-style black olives. On the contrary, 'Manzanilla Cacereña' table olives had a significantly greater TP content, perceived intensity of saltiness (although the same amount of sodium chloride was added to all cans of both olive cultivars) and perceived intensity of cooked defect compared to 'Hojiblanca' table olives. The higher color intensity of 'Hojiblanca' after thermal processing is of major relevance for table olive producers since colored olives are usually more attractive to consumers. Recently, it was reported that 'Manzanilla Cacereña' table olives have a lower color index compared to 'Manzanilla Sevillana' and 'Carrasqueña' (Cabrera-Bañegil, Pérez-Nevado, Montaño, Pleite, & Martín-Vertedor, 2018). Also, the lower intensity of saltiness as well as cooked defect perceived by trained panellists for 'Hojiblanca' table olives may also point out the better suitability of this cultivar for table olive production compared to 'Manzanilla Cacereña', although these latter possessed a greater TP content and so their consumption may have more desirable health-related effects. This significant difference observed in terms of the amount of TP in both olive cultivars may be attributed to genetic factors inherent to each variety (Pérez-Nevado et al., 2018). Furthermore, the results (Table 1 and Fig. 1) also showed that, independently of the olive cultivar, the thermal sterilization applied had a significant effect on the four above mentioned physicochemical and sensory attributes of the studied table olives (P-value < 0.05). A decrease in the color and salty sensation intensity as well as of the TP contents was found on increasing the cumulative lethality of the thermal treatment (i.e., with an increase in the sterilization period, from T1 to T4). The decrease in TP content with an increase of sterilization period observed in this study highlights

Table 1

Two-way ANOVA of the physicochemical parameters and sensory attributes of Californian-style black olives ('Manzanilla Cacereña' and 'Hojiblanca') submitted to different thermal sterilization treatments (T1-T4) of table olives and brine solutions. Results are expressed as mean ± SD of three sample replicates. Different small letters in the same row indicate significant statistical differences between cultivars or thermal treatments (Tukey's Test, p < 0.05).

	'Manzanilla Cacereña'	'Hojiblanca'	*P-value*	T1	T2	T3	T4	*P-value*	*P-value*
	Effect: Cultivar			Effect: thermal treatment					*Interaction "cultivar × thermal treatment"*
Table olives									
Physicochemical parameters									
pH	7.04 ± 0.08^{ns}	7.07 ± 0.08	0.154	7.06 ± 0.10^{ns}	7.02 ± 0.08	7.07 ± 0.09	7.05 ± 0.05	0.610	0.344
Total chlorides (%)	2.06 ± 0.08^{ns}	2.08 ± 0.13	0.622	2.06 ± 0.12^{ns}	2.07 ± 0.09	2.09 ± 0.10	2.08 ± 0.13	0.892	0.775
Total phenol (g GAE/100 g)	3.17 ± 0.78^{b}	2.54 ± 0.42^{a}	< 0.001	3.46 ± 0.37^{d}	3.24 ± 0.61^{c}	2.75 ± 0.40^{b}	1.98 ± 0.11^{a}	< 0.001	< 0.001
Sensory attributes									
Color	7.24 ± 0.66^{a}	8.54 ± 0.61^{b}	< 0.001	8.59 ± 0.63^{b}	8.22 ± 0.84^{b}	7.58 ± 0.48^{a}	7.17 ± 0.89^{a}	< 0.001	< 0.001
Bitter	2.06 ± 0.11^{ns}	2.02 ± 0.28	0.525	2.12 ± 0.12^{ns}	2.05 ± 0.13	2.06 ± 0.17	1.93 ± 0.32	0.084	0.223
Salty	3.23 ± 0.37^{ns}	2.98 ± 0.33	0.001	3.38 ± 0.16^{c}	3.21 ± 0.27^{b}	3.15 ± 0.35^{b}	2.69 ± 0.29^{a}	< 0.001	0.057
Acid	1.95 ± 0.22^{ns}	1.94 ± 0.17	0.881	2.05 ± 0.15^{ns}	1.89 ± 0.17	1.84 ± 0.23	1.98 ± 0.17	0.046	0.934
Cooked defect	3.64 ± 1.90^{ns}	3.10 ± 1.15	< 0.001	1.72 ± 0.16^{a}	2.43 ± 0.16^{b}	3.77 ± 0.46^{c}	5.56 ± 1.03^{d}	< 0.001	< 0.001
Brine solution									
Physicochemical parameters									
pH	7.03 ± 0.10^{ns}	7.04 ± 0.11	0.578	7.04 ± 0.12^{ns}	7.02 ± 0.08	7.06 ± 0.11	7.01 ± 0.10	0.683	0.600
Total chlorides (%)	2.05 ± 0.10^{ns}	2.10 ± 0.16	0.226	2.07 ± 0.10^{ns}	2.09 ± 0.15	2.05 ± 0.16	2.08 ± 0.13	0.898	0.898
Total phenol (g GAE/100 mL)	0.59 ± 0.07^{b}	0.40 ± 0.13^{a}	< 0.001	0.65 ± 0.05^{b}	0.45 ± 0.13^{a}	0.48 ± 0.10^{a}	0.39 ± 0.12^{a}	< 0.001	0.002
Sensory attributes									
Cooked defect	3.77 ± 1.96^{ns}	3.85 ± 1.32	0.508	1.97 ± 0.38^{a}	2.95 ± 0.73^{b}	4.28 ± 0.39^{c}	6.06 ± 0.72^{d}	<0.001	<0.001

ns: not significant.

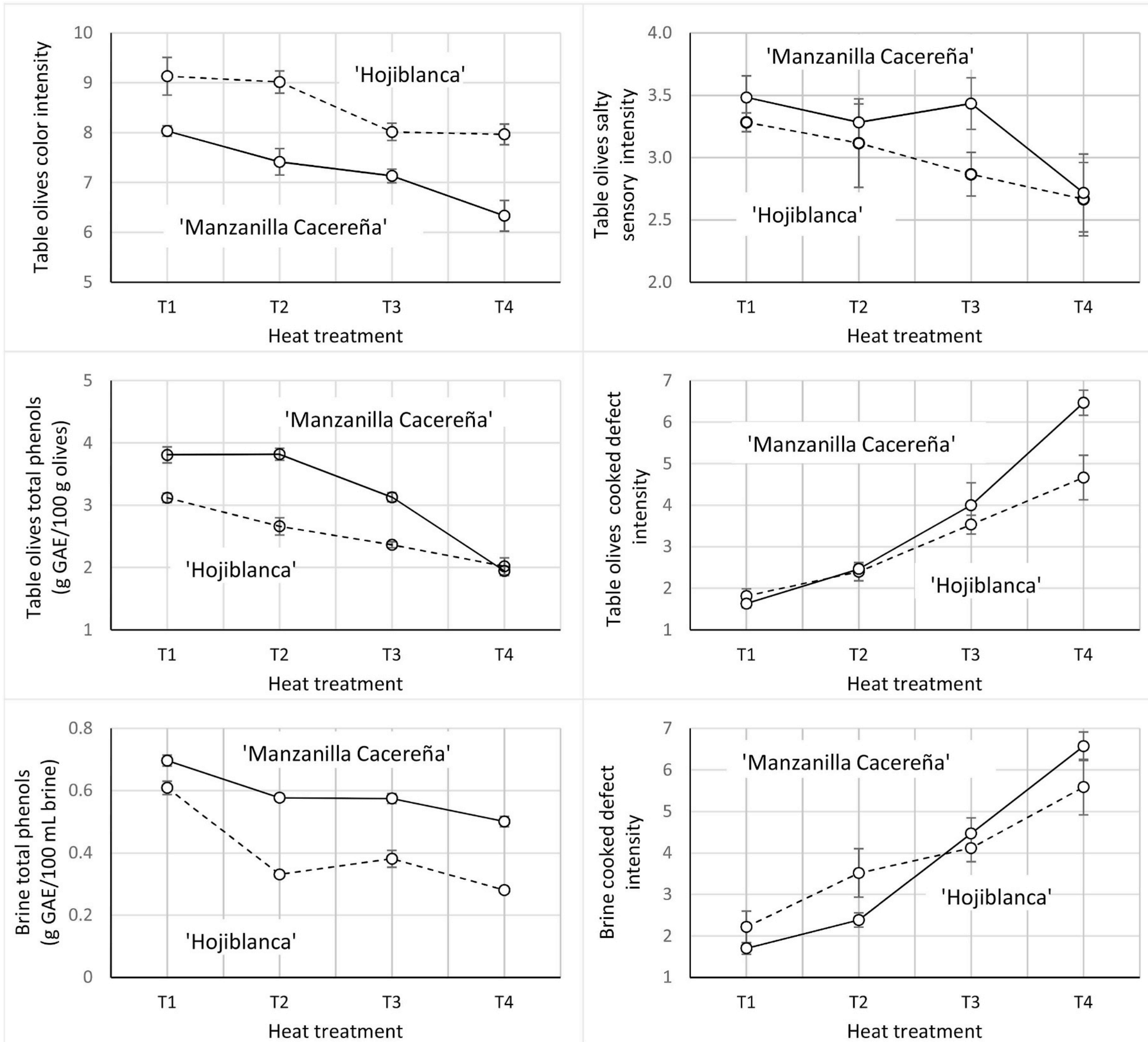

Fig. 1. Physicochemical parameters and sensory attributes of Californian-style black olives ('Manzanilla Cacereña' and 'Hojiblanca') submitted to different thermal sterilization treatments (T1-T4) of table olives and brine solution. Results are expressed as mean ± SD of three sample replicates.

the importance of controlling not only temperature (phenols are easily degraded during the sterilization process due to the high temperature reached) but also the duration of the sterilization process to minimize possible phenols' degradation (Charoenprasert & Mitchell, 2014; Pérez-Nevado et al., 2018) A contrary effect (an increase in sensation intensity with an increase in the thermal treatment duration) could be observed for the cooked defect a trend similar to that previously reported in the literature (Abriouel, Benomar, Gálvez, & Pulido, 2014; Dimou, Panagou, Stoforos, & Yanniotis, 2013). These findings point out an expected negative impact on consumers' preference for purchasing table olives subjected to more aggressive sterilization processes. Indeed, Dimou et al. (2013) and Abriouel et al. (2014) reported that thermal processing is responsible for a deterioration in table olive quality, including color loss and softening of olive tissue. Similar trends and statistically significant effects of olive cultivar or thermal treatment were observed (Table 1 and Fig. 1) on the TP content and cooked defect intensity of the brine solutions. The TP levels in the brine solutions were lower than those found in the respective table olives. The presence of phenols in the brine solutions may be explained by the hydrophilic characteristics of phenols, which justify the tendency of these compounds to migrate from olives to brine (aqueous phase). The migration is influenced by the temperature and pressure attained during the sterilization process that facilitate the translocation of phenols from olives to brine (Lodolini et al., 2019; Pérez-Nevado et al., 2018).

3.2. Qualitative assessment of table olive thermal sterilization treatments using the lab-made potentiometric E-tongue

An LDA-SA-E-tongue model (two discriminant functions explaining 99.9% of the original data variability) was established for each olive cultivar ('Hojiblanca' and 'Manzanilla Cacereña') based on the signals of non-redundant sub-sets of sensors, recorded for the table olive aqueous pastes ('Hojiblanca': S1:1, S1:7, S1:12, S1:14, S1:18, S1:19, S2:1 to S2:3, S2:5 and S2:18; 'Manzanilla Cacereña': S1:1, S1:6, S1:12, S1:20, S2:2, S2:6, S2:14, S2:17, S2:19 and S2:20) or the diluted brine solutions ('Hojiblanca': S1:3, S1:5, S1:7, S1:18, S2:2, S2:5, S2:9, S2:13, S2:16 and S2:20; 'Manzanilla Cacereña': S1:4, S1:7, S1:10, S1:16, S2:17 to S2:20). As can be inferred, the number of sensors as well as the type of sensors used for each olive cultivar differed, which can be attributed to the fact that TP contents and defect intensity varied with the olive cultivar and within each cultivar, depending on whether olive pastes or diluted brine solutions were analyzed. The proposed LDA-SA-E-tongue models allowed the classification of 100% of the original grouped data, for both cultivars and matrices, as can be observed in Fig. 2. Furthermore, satisfactory predictive sensitivities were obtained (LOO-CV procedure), ranging from 92% to 100% ('Hojiblanca': 92% and 96% correct classifications based on the olive paste data or on the diluted brine solution data; 'Manzanilla Cacereña': 100% correct classifications based on the olive paste data or on the diluted brine solution data), a better

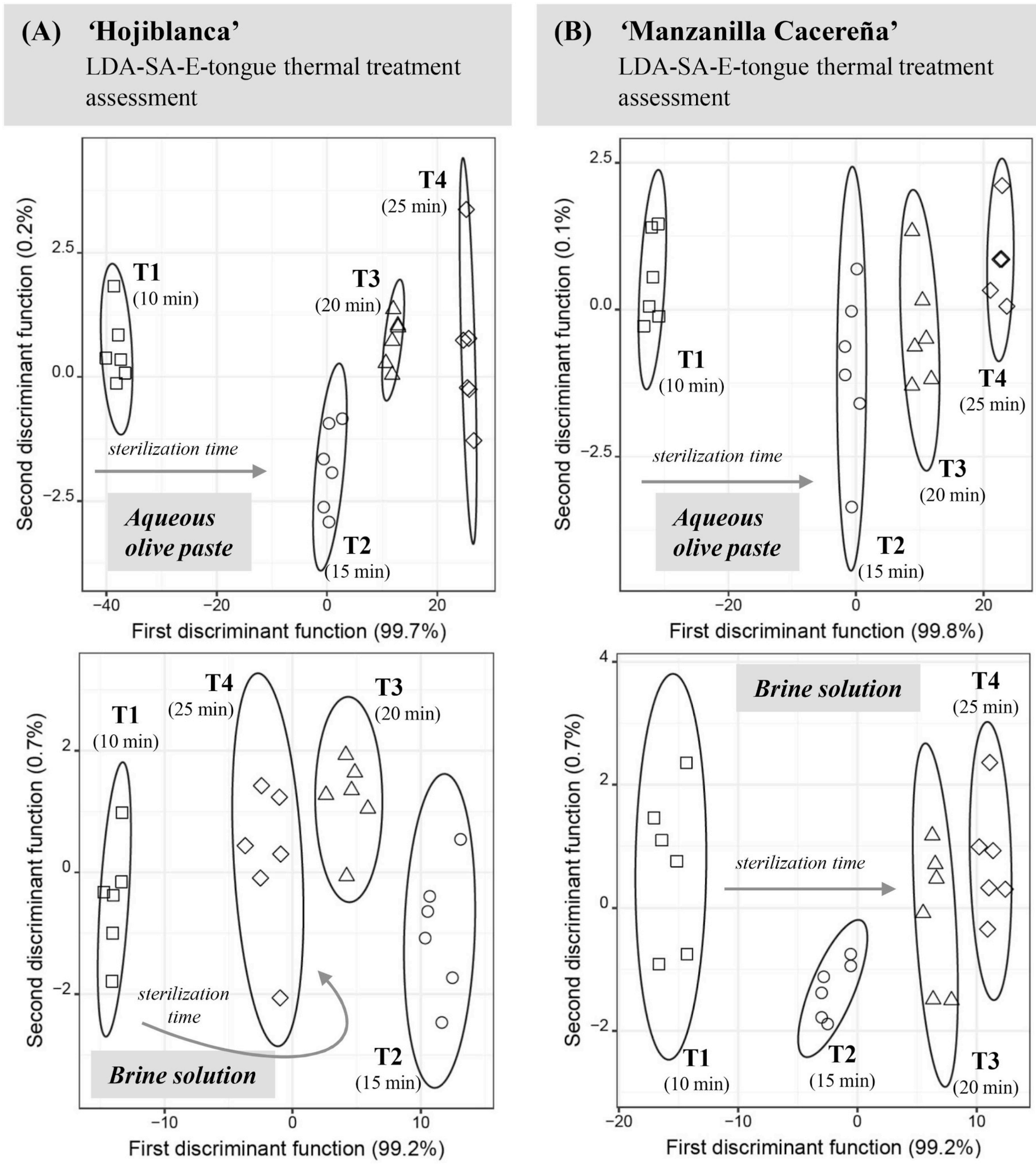

Fig. 2. Discrimination of Californian-style black olives of 'Hojiblanca' (A) or 'Manzanilla Cacereña' (B), according to the thermal sterilization treatment (T1: F_0 = 10 min; T2: F_0 = 15 min, T3: F_0 = 20 min, and T4: F_0 = 25 min, at 121 ± 3 °C) using LDA-SA-E-tongue models established based on the profiles recorded by selected sub-sets of E-tongue sensors during the potentiometric analysis of aqueous olive pastes ('Hojiblanca': S1:1, S1:7, S1:12, S1:14, S1:18, S1:19, S2:1 to S2:3, S2:5 and S2:18; 'Manzanilla Cacereña': S1:1, S1:6, S1:12, S1:20, S2:2, S2:6, S2:14, S2:17, S2:19 and S2:20) or diluted brine solutions ('Hojiblanca': S1:3, S1:5, S1:7, S1:18, S2:2, S2:5, S2:9, S2:13, S2:16 and S2:20; 'Manzanilla Cacereña': S1:4, S1:7, S1:10, S1:16, S2:17 to S2:20).

predictive classification performance being observed for 'Manzanilla Cacereña' table olives. Moreover, for all cases (Fig. 2) the first discriminant function enabled the satisfactory differentiation of the four thermal sterilization processes, it being possible to establish a positive linear correlation between the thermal treatment period (10–25 min) and the centroids of each group ($+0.95 \leq R$-Pearson $\leq +0.98$), except for the diluted brine solutions of 'Hojiblanca' table olives. The predictive performance of the LDA-SA-E-tongue was also checked using the repeated *K*-fold-CV procedure. The results showed that best predictive LDA-SA-E-tongue models were based on the same sensors' signal profiles (acquired during analysis of the aqueous olive pastes or the diluted brine solutions). For aqueous olive pastes mean correct classifications of 89 ± 15% (sensitivity 60–100%) and 94 ± 8% (sensitivity from 75 to 100%) were obtained for 'Hojiblanca' and 'Manzanilla Cacereña', respectively. For the diluted brine solutions, the mean correct classifications ranged from 91 ± 14% (sensitivity from 63 to 100%) to 97 ± 6% (sensitivity from 83 to 100%) for 'Hojiblanca' and 'Manzanilla Cacereña', respectively.

The satisfactory predictive classification performance achieved demonstrates that the E-tongue could be used as practical device for identifying the thermal sterilization treatment period for table olives, allowing a preliminary semi-quantitative assessment of the reduction in the ratio of microorganisms' in terms of their cumulative lethality (F_0 level). The classification capability could be hypothetically attributed

Table 2
Predictive performance of MLRM-SA-E-tongue models regarding the quantification of total phenols (TP) concentrations (determined using the Folin-Ciocalteu method) and cooked defect intensities (assessed by a trained sensory panel) in Californian style black olives from 'Hojiblanca' and 'Manzanilla Cacereña', subjected to different sterilization heat treatments (T1: F_0 = 10 min; T2: F_0 = 15 min, T3: F_0 = 20 min, and T4: F_0 = 25 min, at 121 ± 3 °C).

Olive cultivar	Parameter	Concentration/intensity ranges[a]	E-tongue-MLR-SA models[b]				
			No of sensors[c]	Determination coefficient (R^2)		Root-mean-square errors (*RMSE*)	
				LOO-CV[d]	Repeated *K*-fold-CV[e]	LOO-CV[d]	Repeated *K*-fold-CV[e]
'Hojiblanca'	TP olives(g GAE/100 g)	[1.9, 3.2]	13[f]	0.96	0.96 ± 0.04	0.09	0.10 ± 0.04
	TP brine(g GAE/100 mL)	[0.27, 0.64]	14[g]	0.99	0.98 ± 0.03	0.01	0.02 ± 0.01
	Cooked defect olives(0–10 intensity scale)	[1.5, 5.6]	15[h]	0.99	0.98 ± 0.02	0.11	0.20 ± 0.10
	Cooked defect brine(0–10 intensity scale)	[1.5, 6.2]	13[i]	0.96	0.95 ± 0.04	0.26	0.32 ± 0.15
'Manzanilla Cacereña'	TP olives(g GAE/100 g)	[1.9, 3.9]	10[j]	0.95	0.95 ± 0.03	0.18	0.22 ± 0.07
	TP brine(g GAE/100 mL)	[0.47, 0.72]	14[k]	0.99	0.97 ± 0.03	0.01	0.01 ± 0.01
	Cooked defect olives(0–10 intensity scale)	[1.5, 6.8]	13[l]	0.94	0.96 ± 0.04	0.46	0.51 ± 0.34
	Cooked defect brine(0–10 intensity scale)	[1.5, 7.1]	10[m]	0.97	0.97 ± 0.03	0.32	0.39 ± 0.15

[a] Experimental concentration/defect intensity range levels found in the table olives: total phenols contents assessed using the Folin-Ciocalteu spectrophotometric method; cooked defect intensity assessed by a trained sensory panel using an intensity scale ranging from 0 (sensation not perceived) to 10 (maximum intensity level).
[b] Multivariate linear regression model (MLRM) based on sub-sets of potentiometric sensors, established using the simulated annealing (SA) algorithm, selected among the 40 possible signal profiles obtained with the electronic tongue (E-tongue) during the analysis of the table olives aqueous pastes or the respective brine solutions.
[c] Number of signals included in the MLRM-SA-E-tongue model, selected from the 40 electrochemical signals recorded by E-tongue during analysis of each during the analysis table olives aqueous paste or the respective brine solution.
[d] LOO-CV: leave-one-out cross validation procedure.
[e] Repeated *K*-fold-CV: cross-validation procedure with 4 folds, ensuring that at least 25% of the original data are used for internal validation, and 10 repetitions.
[f] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:1, S1:7, S1:11, S2:1, S2:3, S2:4, S2:7, S2:8, S2:10, S2:11, S2:13, S2:16 and S2:20.
[g] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:6, S1:9, S1:12, S1:19, S1:20, S2:2, S2:6, S2:7, S2:12, S2:13, S2:16, S2:18, S2:19, S2:20.
[h] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:1, S1:4, S1:5, S1:7, S1:19, S2:2, S2:4, S2:5, S2:9, S2:11, S2:12, S2:15, S2:16, S2:19 and S2:20.
[i] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:12, S1:14, S1:20, S2:1, S2:2, S2:8, S2:11, S2:12, S2:13, S2:15, S2:17, S2:18 and S2:19.
[j] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:8, S1:10, S1:16, S1:17, S1:18, S2:8, S2:12, S2:13, S2:19 and S2:20.
[k] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:10, S1:11, S1:12, S2:13, S1:17, S2:1, S2:2, S2:5, S2:7, S2:8, S2:9, S2:13 and S2:15.
[l] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:1, S1:2, S1:8, S1:14, S1:17, S1:18, S1:19, S1:20, S2:1, S2:3, S2:8, S2:9, S2:12, S2:14 and S2:15.
[m] E-tongue sensor signals included in the MLRM-SA-E-tongue model: S1:1, S1:10, S1:11, S1:16, S1:17, S1:18, S2:3, S2:11, S2:14 and S2:19.

to the changes in TP content and cooked defect intensity related to the increase in thermal treatment period. This result is in agreement with the findings of Marx et al. (2017a), who reported the successful discrimination of commercial table olives from different cultivars based on the intensity of sensory defects assessed using a similar E-tongue (print-screen sensor arrays), although the methodology required the simultaneous use of the signals recorded during the analysis of both aqueous olive pastes and brine solutions, leading to a *destructive* analytical approach. On the contrary, in this study, the different thermal treatments studied could be satisfactorily discriminated using the sensor information gathered during analysis of either aqueous olive paste or brine solution, the latter allowing us to foresee a putative *non-destructive* E-tongue evaluation strategy.

3.3. Quantitative analysis of TP content and cooked defect intensity of table olives using the lab-made potentiometric E-tongue

Use of the E-tongue as a quantitative monitoring tool for predicting the influence of the thermal treatments on the TP content (determined by the Folin-Ciocalteu method) and cooked defect intensity (assessed by trained panellists) of 'Hojiblanca' and 'Manzanilla Cacereña'table olives, was further investigated. Two CV variants were used, namely the LOO-CV and repeated *K*-fold-CV procedures (10 repeats × 4 folds). Parameters regarding the goodness of fit of the MLRM-SA-E-tongue models established for the quantification of TP and mean intensity of the perceived cooked sensory defect are reported in Table 2. The number and type of sensors included in each MLRM established are also given in Table 2. The overall R^2 and *RMSE* values (LOO-CV - TP levels: $0.95 \le R^2 \le 0.99$ and $0.01 \le RMSE \le 0.18$ g/100 g or 100 mL, defect intensity: $0.94 \le R^2 \le 0.97$ and $0.11 \le RMSE \le 0.46$; repeated *K*-fold-CV- TP levels: $0.95 \pm 0.03 \le R^2 \le 0.98 \pm 0.03$ and $0.01 \pm 0.01 \le RMSE \le 0.22 \pm 0.07$ g/100 g or 100 mL, defect intensity: $0.95 \pm 0.04 \le R^2 \le 0.98 \pm 0.02$ and $0.20 \pm 0.10 \le RMSE \le 0.51 \pm 0.34$) allowed verification of the satisfactory performance of the potentiometric E-tongue approach for quantifying TP as well as the median intensity of the cooked defect perceived in 'Hojiblanca' and 'Manzanilla Cacereña' table olives, based on analysis of the aqueous olive paste or diluted brine solutions. Fig. 3 showed the satisfactory results achieved for the repeated *K*-fold-CV variant. The results are in agreement with those previously reported by Marx et al. (2017b,c) for assessment of the positive and negative sensory sensations perceived in commercial table olives and by Rodrigues et al. (2019) for monitoring the impact of the debittering process on the chemical and sensory sensations of traditional stoned green table olives. It should be remarked that Marx et al. (2017b, 2017c) used the potentiometric signals recorded for both aqueous olive pastes and brine solutions, which poses a practical limitation compared to the analytical methodology applied in the present study, which only required the use of either the olive paste data or the brine solution data, allowing the implementation of a *non-destructive* analysis strategy. Indeed, the superior performance now achieved may be attributed to the larger superficial surface and greater thickness of the sensor lipid membranes in

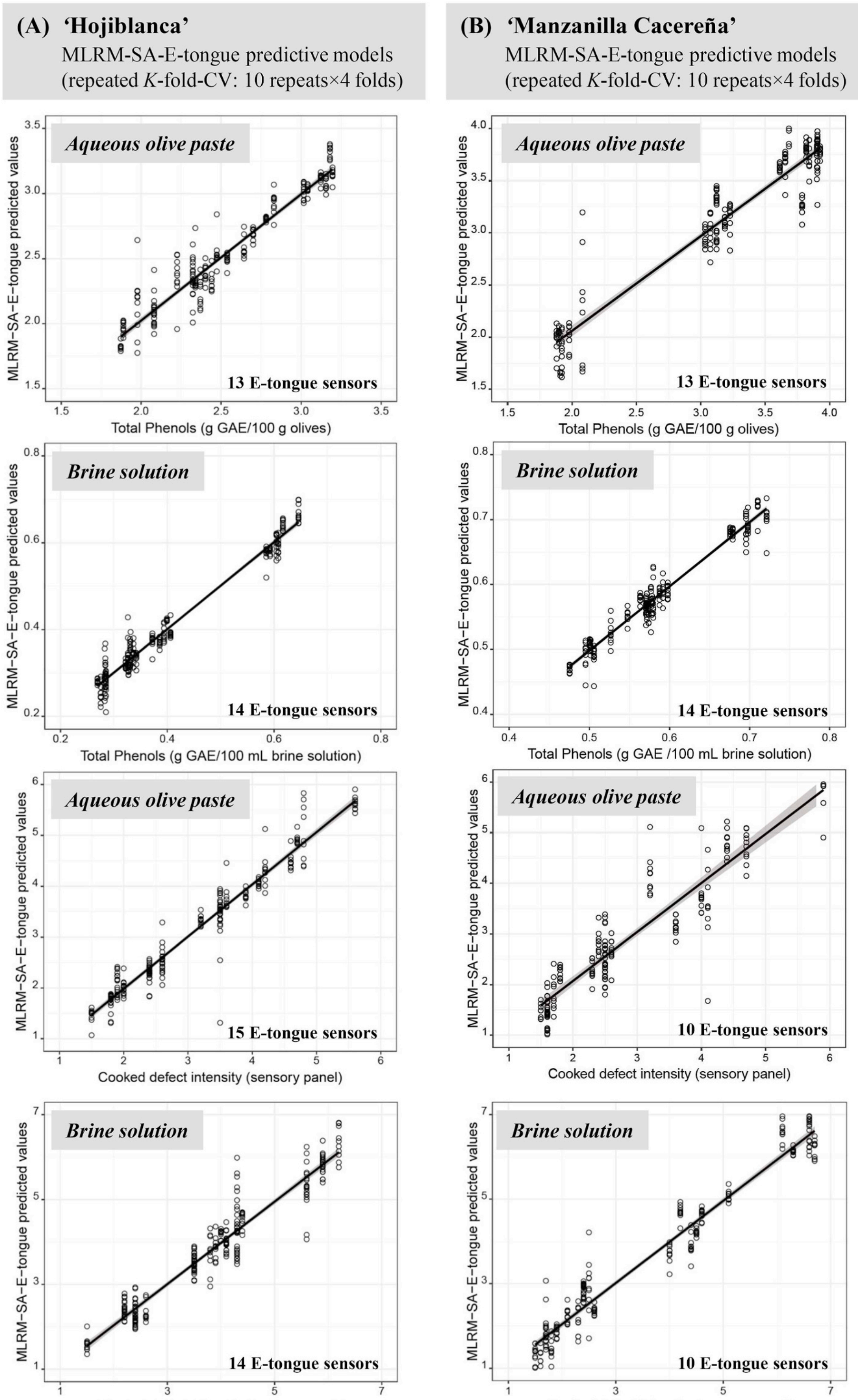

Fig. 3. Total phenols (TP) concentrations and median cooked defect intensity predicted using MLRM-SA-E-tongue models (repeated *K*-fold-CV variant: 10 repeats × 4 folds), established based on different sub-sets of sensors (10–15 E-tongue sensors) selected using the SA algorithm, versus the experimental data determined using the Folin-Ciocalteu method and a trained sensory panel, during the analysis of Californian-style black olives (aqueous olive pastes and respective diluted brine solutions), subjected to different thermal sterilization treatments (T1: F_0 = 10 min; T2: F_0 = 15 min, T3: F_0 = 20 min, and T4: F_0 = 25 min, at 121 ± 3 °C) versus for: (A) 'Hojiblanca'; and, (B) 'Manzanilla Cacereña'.

the E-tongue used in the present work compared with those incorporated in the E-tongue device used by Marx et al. (2017b, 2017c). The results allow verification of the hypothesis that a greater contact area and thickness would increase the potentiometric signal sensitivity to the chemical compounds responsible for both the positive and negative sensory sensations perceived in table olives, after the different thermal sterilization treatments used.

Finally, application of the MLRM-SA-E-tongue models as a complementary accurate analytical approach for assessing the TP level or cooked defect intensity of table olives was further studied (Roig & Thomas, 2003a; 2003b). Thus, single linear regressions were established between the TP concentration or cooked defect intensity predicted by the MLRM-SA-E-tongue models and the related experimental data assessed by standard methods (Folin–Ciocalteu and sensory panel, respectively), it being evaluated if the slope and intercept values were statistically equal to those of a perfect linear fit (one and zero, respectively). The parameters of the single linear regressions (R^2, slope and intercept values and the respective 95% confidence intervals) for LOO-CV and repeated *K*-fold-CV techniques are given in Table 3. The results demonstrate that, at 5% significance level, the slope and

Table 3
Parameters of the single linear regressions established between the values of concentration/intensity levels predicted by MLR-SA-E-tongue models (LOO-CV and repeated *K*-fold-CV) and the respective experimental total phenols (TP) concentrations/cooked defect intensities (quantified using the Folin-Ciocalteu spectrophotometric method or a trained sensory panel, respectively): coefficient of determination (R^2); slopes, intercept values and respective confidence intervals (CI) at 95%.

Olive cultivar	Parameter	LOO-CV[a]					Repeated K-fold-CV[b]				
		R^2	Slope	Slope CI[a]	Intercept	Intercept CI[b]	R^2	Slope	Slope CI[a]	Intercept	Intercept CI[b]
'Hojibl0061nca'	TP olives	0.96	0.98	[0.89, 1.07]	0.05	[−0.18, 0.29]	0.92	0.98	[0.94, 1.01]	0.07	[−0.03, 0.16]
	TP brine	0.99	0.99	[0.95, 1.05]	0.001	[−0.02, 0.02]	0.94	1.01	[0.98, 1.04]	−0.004	[−0.02, 0.01]
	Cooked defect olives	0.99	1.00	[0.96, 1.04]	−0.0004	[−0.14, 0.14]	0.88	1.01	[0.96, 1.05]	−0.04	[−0.20, 0.12]
	Cooked defect brine	0.96	0.97	[0.89, 1.05]	0.103	[−0.23, 0.44]	0.90	0.97	[0.93, 1.01]	0.10	[−0.07, 0.26]
'Manzanilla Cacereña'	TP olives	0.95	1.00	[0.90, 1.10]	−0.03	[−0.36, 0.31]	0.90	0.99	[0.96, 1.04]	−0.02	[−0.16, 0.11]
	TP brine	0.99	0.99	[0.94, 1.03]	0.004	[−0.02, 0.03]	0.95	0.99	[0.96, 1.02]	0.003	[−0.01, 0.02]
	Cooked defect olives	0.94	0.92	[0.82, 1.02]	0.20	[−0.20, 0.60]	0.87	0.97	[0.92, 1.01]	0.11	[−0.08, 0.30]
	Cooked defect brine	0.97	0.99	[0.92, 1.06]	0.02	[−0.28, 0.32]	0.95	1.01	[0.98, 1.03]	−0.03	[−0.15, 0.09]

[c] 95% slope confidence interval.
[d] 95% intercept confidence interval.
[a] LOO-CV (leave-one-out cross-validation).
[b] Repeated *K*-fold-CV (4 folds × 10 repeats).

intercept values were statistically equal to the expected theoretic values. These results confirm that the MLRM-SA-E-tongue strategy could be used for monitoring the effect of thermal treatment on the TP content and on the cooked defect intensity during the sterilization of Californian-style black olives. This finding strengthens the concept that potentiometric taste sensor-based devices could be used as a complementary/alternative technique to the standard Folin–Ciocalteu spectrophotometric method or trained sensory panels, for assessing, respectively, the changes in TP level and in perception level of the intensity of cooked defect in table olives subjected to typical industrial heat sterilization processes.

4. Conclusions

The application of thermal treatments caused a clear varietal difference. In fact, 'Manzanilla Cacereña' olives presented a lower color intensity, higher TP content, greater salty intensity and greater cooked defect intensity than 'Hojiblanca' olives, this latter cultivar apparently being more adequate for producing Californian-style black olives. Besides that, increasing the thermal sterilization duration resulted in a decrease of the color intensity, TP content and cooked defect sensation, pointing out the advantages of using shorter sterilization periods for obtaining table olives with better quality characteristics. Finally, the E-tongue was shown to be a fast and useful tool for discriminating table olives according to the thermal sterilization process applied. Moreover, the use of thicker sensor lipid membranes with a larger superficial contact surface enhanced the sensitivity of the potentiometric device, allowing a satisfactory qualitative and quantitative assessment of chemical-sensory attributes based only on brine analysis, leading to a non-destructive analytical technique for the quality analysis of table olives.

Compliance with ethics requirements

This research does not include any experiment with animal and/or human subjects.

CRediT authorship contribution statement

Daniel Martín-Vertedor: Conceptualization, Formal analysis, Writing - original draft, Writing - review & editing. **Nuno Rodrigues:** Conceptualization, Writing - original draft, Writing - review & editing. **Ítala M.G. Marx:** Funding acquisition, Formal analysis, Writing - review & editing. **Ana C.A. Veloso:** Funding acquisition, Writing - review & editing. **António M. Peres:** Conceptualization, Formal analysis, Writing - original draft, Writing - review & editing. **José Alberto Pereira:** Conceptualization, Formal analysis, Writing - original draft, Writing - review & editing.

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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