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Sensory intensity assessment of olive oils using an electronic tongue

Ana C.A. Veloso [a,b], Luís G. Dias [c,d], Nuno Rodrigues [e], José A. Pereira [e], António M. Peres [f,*]

[a] Instituto Politécnico de Coimbra, ISEC, DEQB, Rua Pedro Nunes, Quinta da Nora, 3030-199 Coimbra, Portugal
[b] CEB-Centre of Biological Engineering, University of Minho, Campus de Gualtar, 4710-057 Braga, Portugal
[c] Escola Superior Agrária, Instituto Politécnico de Bragança, Campus Santa Apolónia, 5301-855 Bragança, Portugal
[d] CQ-VR, Centro de Química – Vila Real, University of Trás-os-Montes e Alto Douro, Apartado 1013, 5001-801 Vila Real, Portugal
[e] CIMO-Mountain Research Centre, Escola Superior Agrária, Instituto Politécnico de Bragança, Campus Santa Apolónia, 5301-855 Bragança, Portugal
[f] LSRE-Laboratory of Separation and Reaction Engineering-Associate Laboratory LSRE-LCM, Escola Superior Agrária, Instituto Politécnico de Bragança, Campus Santa Apolónia, 5301-855 Bragança, Portugal

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ABSTRACT

Olive oils may be commercialized as intense, medium or light, according to the intensity perception of fruitiness, bitterness and pungency attributes, assessed by a sensory panel. In this work, the capability of an electronic tongue to correctly classify olive oils according to the sensory intensity perception levels was evaluated. Cross-sensitivity and non-specific lipid polymeric membranes were used as sensors. The sensor device was firstly tested using quinine monohydrochloride standard solutions. Mean sensitivities of 14 ± 2 to 25 ± 6 mV/decade, depending on the type of plasticizer used in the lipid membranes, were obtained showing the device capability for evaluating bitterness. Then, linear discriminant models based on sub-sets of sensors, selected by a meta-heuristic simulated annealing algorithm, were established enabling to correctly classify 91% of olive oils according to their intensity sensory grade (leave-one-out cross-validation procedure). This capability was further evaluated using a repeated K-fold cross-validation procedure, showing that the electronic tongue allowed an average correct classification of 80% of the olive oils used for internal-validation. So, the electronic tongue can be seen as a taste sensor, allowing differentiating olive oils with different sensory intensities, and could be used as a preliminary, complementary and practical tool for panelists during olive oil sensory analysis.

1. Introduction

Different olive oils are commercially available and their prices depend on the olive oil quality grade (extra-virgin, EVOO; virgin or lampante), geographical origin and production procedure. Olive oils may also be labeled as intense, medium or light (mild), based on the sensory intensity perception of positive olfactory and gustatory–retronasal attributes. For this, olive oils fruitiness, bitterness and pungency levels are evaluated by trained sensory panelists, which besides being a time-consuming task, limits the maximum number of samples that may be assessed per day. Each attribute is evaluated using a quantitative scale (ranging from 0 (minimum) to 10 (maximum)), following the regulations of the International Olive Council [1,2].

Olive oils are highly appreciated by consumers and play a key role in several diets, due to the organoleptic attributes and the health benefits. The economic value of olive oils make this food product very prone to fraud, including mislabeling of olive oil commercial category, geographical or olive cultivar origin [3–7]. So, several gas-, liquid- and mass-spectrometry chromatography, DNA and spectroscopy based methods have been developed to assess olive oil quality and authenticity as well as to detect possible adulterations [3,5,6,8–16]. Electrochemical sensors have also been extensively used, including electronic noses and electronic tongues (E-tongues), individually or in combination, mainly with the aim of identifying possible adulterations or classifying olive oils according to quality level, geographical origin or olive cultivar [16–28]. Recently, a "magnetic tongue" was used to quantify minor compounds of EVOO that are related to the sensory attributes [29]. However, the potential of electrochemical devices to classify olive oils based on the intensity perception of positive sensory attributes was never investigated. The availability of fast and cost-effective analytical techniques for preliminary verification of the compliance of this high quality EVOO qualitative classification (i.e., intense, medium or light olive oils) is a challenge task and pertinent issue since (i) nowadays, consumers increasingly require this kind of olive oil label information; (ii) olive oil producers are using this information as an additional organoleptic information in their products to increase its commercial value; and, (iii) the maximum number of olive oils that can be evaluated per day by a trained

* Corresponding author. Fax: +35 1273325405.
E-mail address: peres@ipb.pt (A.M. Peres).

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sensory panel is quite limited. So the capability of a potentiometric E-tongue to classify monovarietal EVOO according to the perceived sensory intensity level (i.e., light, medium or intense) was evaluated. Since olive oils are non-conductive and high viscous, the potentiometric signals were recorded in ethanol–water extracts, which are rich in polar compounds that are related with bitterness, pungency and astringency sensations. The olive oil samples analyzed were collected during two crop years (2012 and 2013) and belong to 11 Spanish single-cultivar EVOO: *cvs.* Arbequina, Arbosana, Arroniz, Cornicabra, Frantoio, Hojiblanca, Manzanilla, Picual, Redondilla, Royuela and Zorzal. The classification performance of the E-tongue was assessed using linear discriminant analysis (LDA) based on the most informative potentiometric sensor signals selected using a meta-heuristic simulated annealing (SA) algorithm.

2. Materials and methods

2.1. Reagents

In all assays, deionized type II water was used. Folin-Ciocalteau, sodium carbonate, n-hexane, methanol and ethanol were of analytical grade (Panreac, Barcelona). Quinine monohydrochloride dihydrate was purchased to Sigma-Aldrich (p.a., minimum purity $\geq$ 90%). For E-tongue arrays construction, all reagents were from Fluka (minimum purity $\geq$97%): plasticizers (bis(1-butylpentyl) adipate, dibutyl sebacate, 2-nitrophenyl-octylether, tris(2-ethylhexyl)phosphate and dioctyl phenylphosphonate) and additives (octadecylamine, oleyl alcohol, methyltrioctylammonium chloride and oleic acid). High molecular weight polyvinyl chloride was used as the supporting polymer (Fluka).

2.2. Olive oil samples

Eleven monovarietal Spanish EVOOs (cvs. Arbequina, Arbosana, Arroniz, Cornicabra, Frantoio, Hojiblanca, Manzanilla, Picual, Redondilla, Royuela and Zorzal), produced at the north of Spain (Valladolid region), were studied. In total, 88 different samples of single-cultivar EVOO were obtained directly from olive oil certified producers during 2012–2013 (Table 1). Olive oils were packed and stored in dark at −20 °C in a 24-h period after their production in olive mills with a two-phase extraction process and kept in those conditions until further analysis.

2.3. Olive oil total phenolic content

Total phenolic contents were determined as described by Capannesi et al. [30] with some modifications. A mass of 2.5 g of olive oil was diluted with n-hexane (1:1) and extracted three times with 2.5 mL of methanol/water (80:20; v/v). The mixture was then centrifuged (5 min at 2600 g). To 1 mL of the combined extract it was added 1 mL of Folin-Ciocalteau reagent, 1 mL of Na_2CO_3 solution (7.5%) and 7 mL of deionized water, in order to obtain a final volume of 10 mL. After homogenization, the mixture was stored overnight and spectrophotometrically analyzed (λ=765 nm). For quantification purposes a calibration curve between the measured absorbance and the concentration of caffeic acid in methanol was established (dynamic concentration range: 0.04–0.18 mg/mL). A new calibration curve was established each day before olive oil analysis (R^2-Pearson $\geq$ 0.996). The final results were expressed as mg of caffeic acid equivalents per kg of olive oil (mg CAE/kg).

2.4. Olive oil sensory analysis

Olive oil samples were subjected to sensory assessment following the methods and standards adopted by the International Olive Council (COI), namely COI/T.20/Doc. no 15/Rev. 6 [1] and COI/T.30/Doc. no 17 [2]. Each sample was subjected to the judgment of four trained panel members that classified the samples according to olfactory sensations, gustatory – retronasal sensations and final olfactory–gustatory sensations. Based on these evaluations and following the COI regulations, monovarietal EVOO samples were classified as intense, medium or light/mild olive oils according to the intensity perception of four positive attributes (olfactory and gustatory olive fruitiness; gustatory–retronasal bitterness and pungency). This labeling classification is based on the median

Table 1
Details of the monovarietal EVOO samples collected in Valladolid region (Spain): olive cultivar, production year, intensity perception level of positive sensory attributes.

Monovarietal EVOO	Production year	Number of samples	Intensity perception of positive sensory attributes (Intenational Olive Council, 2013) Gustatory-retronasal sensations				Group
			Olive fruitiness		Bitterness	Pungency	
Arbequina	2012	2	Intense	Intense	Intense	Intense	IIII
		3	Intense	Intense	Medium	Intense	IIMI
		7	Intense	Intense	Medium	Medium	IIMM
	2013	4	Intense	Intense	Medium	Intense	IIMI
		8	Intense	Intense	Medium	Medium	IIMM
Arbosana	2012	4	Intense	Intense	Intense	Intense	IIII
	2013	4	Intense	Intense	Medium	Intense	IIMI
Arroniz	2012	4	Intense	Intense	Medium	Intense	IIMI
Cornicabra	2012	4	Intense	Intense	Medium	Intense	IIMI
	2013	4	Intense	Intense	Medium	Medium	IIMM
Frantoio	2012	4	Intense	Intense	Medium	Intense	IIMI
	2013	4	Intense	Intense	Medium	Intense	IIMI
Hojiblanca	2012	4	Intense	Intense	Intense	Intense	IIII
	2013	4	Intense	Intense	Intense	Intense	IIII
Manzanilla	2012	4	Intense	Intense	Light	Light	IILL
Picual	2012	4	Intense	Intense	Medium	Intense	IIMI
	2013	4	Intense	Intense	Medium	Intense	IIMI
Redondilla	2012	4	Intense	Intense	Medium	Medium	IIMM
	2013	4	Intense	Intense	Medium	Medium	IIMM
Royuela	2013	4	Intense	Intense	Intense	Intense	IIII
Zorzal	2013	4	Intense	Intense	Medium	Intense	IIMI

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