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A taste sensor device for unmasking admixing of rancid or winey-vinegary olive oil to extra virgin olive oil

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ABSTRACT

Electrochemical sensor devices have gathered great attention in food analysis namely for olive oil evaluation. The adulteration of extra-virgin olive oil with lower-grade olive oil is a common worldwide fraudulent practice, which detection is a challenging task. The potentiometric fingerprints recorded by lipid polymeric sensor membranes of an electronic tongue, together with linear discriminant analysis and simulated annealing meta-heuristic algorithm, enabled the detection of extra-virgin olive oil adulterated with olive oil for which an intense sensory defect could be perceived, specifically rancid or winey-vinegary negative sensations. The homemade designed taste device allowed the identification of admixing of extra-virgin olive oil with more than 2.5% or 5% of rancid or winey-vinegary olive oil, respectively. Predictive mean sensitivities of 84 ± 4% or 92 ± 4% and specificities of 79 ± 6% or 93 ± 3% were obtained for rancid or winey-vinegary adulterations, respectively, regarding an internal-validation procedure based on a repeated K-fold cross-validation variant (4 folds × 10 repeats, ensuring that the dataset was forty times randomly split into 4 folds, leaving 25% of the data for validation purposes). This performance was satisfactory since, according to the legal physicochemical and sensory analysis, the intentionally adulterated olive oil with percentages of 2.5–10%, could still be commercialized as virgin olive oil. It could also be concluded that at a 5% significance level, the trained panelists could not distinguish extra-virgin olive oil samples from those adulterated with 2.5% of rancid olive oil or up to 5% of winey-vinegary olive oil. Thus, the electronic tongue proposed in this study can be foreseen as a practical and powerful tool to detect this kind of worldwide common fraudulent practice of high quality olive oil.

1. Introduction

Olive oil quality classification as extra-virgin (EVOO), virgin (VOO) or lampante (LOO) olive oil is regulated by the European Union Commission (EU No 61/2011, 2011; EU No 1348/2013, 2013). These regulations take into account the legal levels defined for physico-chemical parameters (e.g., free acidity, peroxide value, UV extinction coefficients and alkyl esters content), as well as, for positive and negative sensory sensations such as, the perception and the intensity of fruity positive attribute and the presence/absence of sensory defects (e.g., fusty, musty, rancid, winey-vinegary) (Borràs et al., 2015, 2016a, 2016b; Di Serio et al., 2017). Olive oil is highly appreciated by worldwide consumers due to the recognized health and nutritional benefits. Adulteration, frauds and mislabeling of olive oil have become a worldwide phenomenon leading to the decrease of the confidence of consumers (Jolayemi et al., 2017). Thus, different analytical techniques have been developed to detect olive oil adulterations (e.g., MALDI-TOF/MS technique; mid infrared, Raman, fluorescence or visible spectroscopy; DNA-targeted approaches; ion mobility spectrometry; nuclear magnetic resonance; dielectric technique; ultrasounds technique; gas chromatography; etc.), namely to identify and/or quantify the addition of other vegetable oils like camellia, canola, corn, grapeseed, hazelnut,

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peanut, rapeseed, soya, sesame, soybean and sunflower oils (De Melo Milanez and Pontes, 2015; Sun et al., 2015; Alouache et al., 2016; Jabeur et al., 2016; Kalaitzis and El-Zein, 2016; Nigri and Oumeddour, 2016; Mu et al., 2016; Rashvand et al., 2016; Srigley et al., 2016; Farley et al., 2017; Georgouli et al., 2017; Jergović et al., 2017; Liu et al., 2017; Ok, 2017; Philippidis et al., 2017; Santos et al., 2017; Uncu et al., 2017) or the admixture of lower quality or refined olive oils (Nigri and Oumeddour, 2016; Jergović et al., 2017). Although EVOO have a long history of economic adulteration, its detection still is a challenging task due to the diverse composition of cultivars and the limitations of existing detection methods (Ou et al., 2015; Srigley et al., 2016). The broad use of sensor-based devices, like electronic noses (E-noses) or electronic tongues (E-tongues), for olive oil sensory evaluation or olive oil discrimination based on the olive cultivar and geographical origin has been recently reviewed by Peris and Escuder-Gilabert (2016) and Valli et al. (2016). The literature survey clearly point out the limited number of works reporting the successful use of E-noses (Oliveros et al., 2002; Mildner-Szkudlarz and Jeleń, 2008, 2010; Lerma-García et al., 2010; Santonico et al., 2015) to detect olive oil adulteration with other vegetable oils or lower quality olive oils (possessing or not common sensory defects), as well as the scarce use of voltammetric E-tongues (Apetrei and Apetrei, 2014; Santonico et al., 2015). Recently, the use of a pontentiometric E-tongue device comprising cross-sensitivity lipid polymeric membranes, has demonstrated to be a practical and helpful taste sensor tool for olive oil analysis (Dias et al., 2014, 2016; Veloso et al., 2016, 2018; Slim et al., 2017; Souayah et al., 2017). It was previously reported by Marx et al. (2017b) and Slim et al. (2017) the capability of this type of E-tongue to provide quantitative potentiometric responses towards aldehydes, alcohols and esters compounds that mimic positive olive oil sensory attributes namely, 4-hydroxy-3-methoxybenzaldehyde (vanilla sensation), hexyl acetate (sweet, green, grassy, fruity or apple sensations), (Z)-hex-3-en-1-ol (green leaves or banana sensations), (E)-hex-2-enal (green, almonds or apple sensations), (Z)-hex-3-enyl acetate (fruity or green leaves sensations), citric and tartaric acids (acid sensation), caffeine and quinine (bitter sensations) and sodium or potassium chloride (salty sensation). On the other hand, for negative sensations, Marx et al. (2017a) also described the quantitative responses towards n-butyric acid (butyric defect), 2-mercaptoethanol (putrid defect) and cyclohexanecarboxylic acid (zapateria defect). The sensing mechanism is dependent on the non-uniform hydrophilicity of the lipid membranes and on the ionic environment at the proximity of the membrane surface. Thus, the measured electric potential depends on the membrane surface-charge density changes, and on its permeability to ions altered by the physical adsorption of non-electrolytes compounds (Iiyama et al., 1986; Kurihara et al., 1986; Hayashi et al., 1989). Recently Veloso et al. (2018) reported the capability of using an E-tongue device to classify olive oil according to the main sensory defect perceived. In this work, a pontentiometric E-tongue device was applied, for the first time, for detecting intentionally-adulterated EVOO with known percentages of rancid or winey-vinegary LOO (LOO-R or LOO-WV), which sensory defect and intensities were assessed by trained panelists.

2. Materials and methods

2.1. Olive oil samples, physicochemical and sensory analysis

Olive oils, produced from olives of Arbequina variety, were kindly supplied by a local olive oil producer of the Trás-os-Montes region (Macedo de Cavaleiros, Portugal). Fifteen liters of a high quality olive oil (EVOO) and ten liters of two types of low quality olive oil (LOO) were used. The LOO samples were intentionally chosen after ensuring that rancid (LOO-R, 5 Ls) or winey-vinegary (LOO-WV, 5 Ls) negative sensations could be easily perceived by a sensory panel due to their high intensities. For the experiment, intentionally-adulterated EVOO olive oil samples were prepared and used, obtained by adding EVOO samples with pre-established volumes of LOO, resulting in volumetric adulterations (v/v) of the EVOO with 2.5%, 5%, 10%, 20% and 40% of LOO-R or LOO-WV. In total, 6 glass amber bottles of 250 mL each, were prepared for each adulteration level (2.5–40%, for each organoleptic defect, plus the negative (EVOO, i.e., 0% of adulteration) and positive (LOO-R or LOO-WV) controls. The established volumetric percentage levels took into account the fact that a 10% level of adulteration is high enough to be economically profitable but low enough to pass undetected (Srigley et al., 2016). Olive oil samples (EVOO, LOO-R, LOO-WV and respective adulterated olive oil) were kept in amber bottles protected from the direct light exposition during 2 weeks before being used. To check the quality of the EVOO, LOO-R, LOO-WV and the intentionally-adulterated olive oil, all samples were subjected to physicochemical and sensory analysis, following the EU standard methods (EU No 61/2011, 2011; EU No 1348/2013, 2013). Five physicochemical quality parameters were evaluated: free acidity (FA, in% oleic acid), the peroxide value (PV, in mEq O_2/kg) and the specific coefficients of extinction at 232 nm and 270 nm (K_{232} and K_{270} and ΔK). From each olive oil sample (n = 6), three independent sub-samples were collected and all physicochemical and chemical assays were carried out in triplicate. Each sample was also evaluated by eight trained panelists from the olive oil sensory panel of the School of Agriculture of the Polytechnic Institute of Bragança (Portugal), which was instructed by the panel leader about the type of defect that might be perceived or not in each sample (i.e., rancid or winey-vinegary sensations). The intensity of the positive or negative attributes perceived were graded according to an intensity scale ranging from 0 (no sensory sensation perceived) to 10 (maximum intensity of the sensory sensation perceived). Furthermore, for the final olive oil' quality grade classification the median intensities were used. The quality grade of the samples (EVOO, LOO and adulterated olive oil) was set considering the physicochemical levels and the sensory data (EU No 61/2011, 2011; EU No 1348/2013, 2013; IOC, 2013, 2014): EVOO (FA ≤ 0.8% oleic acid, PV ≤ 20 mEq O_2/kg, $K_{232} \leq 2.50$, $K_{270} \leq 0.22$, $\Delta K \leq 0.01$; fruity median intensity greater than 0 and median intensity of defects equal to 0); VOO (FA ≤ 2.0% oleic acid, PV ≤ 20 mEq O_2/kg, $K_{232} \leq 2.60$, $K_{270} \leq 0.25$, $\Delta K \leq 0.01$; fruity median intensity greater than 0 and median intensity of defects greater than 0 and lower than 3) or LOO (in the other cases). All assays were performed at the laboratories of the School of Agriculture – Polytechnic Institute of Bragança (Portugal).

2.2. E-tongue

2.2.1. E-tongue device and set-up

The E-tongue multi-sensor device (Fig. 1) included two homemade print-screen potentiometric arrays covered with an acrylic resin (PLASTIK 70) for ensuring a waterproof surface. As previously described (Dias et al., 2015; Veloso et al., 2018), each polyvinyl chloride (PVC) board (3 cm × 12 cm) had 20 wells (3.6 mm of diameter and 0.3 mm of thickness), where 20 cross-sensitivity lipid polymeric membranes were applied, using a drop-by-drop technique. The polymeric membranes had different combinations of 4 lipid additives (~3%: octadecylamine, oleyl alcohol, methyltrioctylammonium chloride or oleic acid), 5 plasticizers (~32%: bis(1-butylpentyl) adipate, dibutyl sebacate, 2-nitrophenyl-octylether, tris(2-ethylhexyl)phosphate or dioctyl phenylphosphonate) and PVC (~65%), which were used as chemical sensors (Fluka, minimum purity ≥97%). Even if the two E-tongue arrays comprised sensor membranes with the same lipid additive/plasticizer/PVC mixture and with the same relative composition, they showed different electrochemical properties, which could be attributed to the formation of inhomogeneous membranes with different physical properties (e.g., different membrane transparency levels and porosity leading to different adsorption phenomena and surface chemical reactions, which may lead to deviations in sensors' readings). Therefore, it was considered that the device comprised 40 independent sensors instead of assuming a set of 20 sensor-sensor replica membranes. At the

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