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ORIGINAL PAPER

# Influence of household conditions on the shelf-life and polyphenolic-related health claim of Cobrançosa extra virgin olive oil

Siwar Alimi[1,2] · Nuno Rodrigues[1] · Ana C. A. Veloso[3] · Rebeca Cruz[4] · Maissa Khemakhem[2] · José Alberto Pereira[1] · Susana Casal[4] · António M. Peres[1]

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## Abstract

This study examines how typical household conditions after bottle opening affect the physicochemical, sensory, and bioactive properties of cv. Cobrançosa extra virgin olive oil (EVOO), attempting to define kinetic models to predict the shelf-life (SL) during domestic use. For 9 weeks, EVOO amber glass bottles (750 mL), exposed to light ($n=5$) or darkness ($n=5$), at 18 ± 2 ºC, were opened/shaken daily to simulate household use, with oil removed weekly. In light-exposed samples, the peroxide value (PV) imposed EVOO declassification at week five, with intense rancidity at week eight (≥ 3.5), rendering the oil unsuitable for consumption. On the contrary, light-protected oils had only a downgrade to virgin olive oil due to a $K_{232}$ rise. Acidity was preserved, as was the health claim supported by tyrosol/hydroxytyrosol polyphenols. Kinetic models (zero-, first-, and second-order) supported on the oxidation indicators (PV, $K_{232}$, or $K_{268}$), allowed determining reaction rates by linear regression (correlation coefficients: 0.942 to 0.997). For light-exposed oils, PV was the most reliable indicator of SL, predicting from a second-order TRUL model a preservation of the EVOO grade for 35 ± 2 days, in agreement with the experimental SL (28–35 days). For light-protected oils, $K_{232}$ was the most accurate SL indicator, predicting a SL of 49 ± 4 days using a zero-order TRUL model, consistent with the experimental SL (49–56 days). The models were validated using SL literature data from cvs. Arbequina, Istarska Bjelica, and Buža olive oils, confirming their applicability to various cultivars and highlighting oxidation's role, particularly photo-oxidation, in EVOO degradation during domestic use.

✉ Susana Casal
sucasal@ff.up.pt

✉ António M. Peres
peres@ipb.pt

1 CIMO, LA SusTEC, Instituto Politécnico de Bragança, Campus de Santa Apolónia, 5300-253 Bragança, Portugal

2 Université Libre de Tunis, 32 Bis Av. Kheireddine Pacha, 1002 Tunis, Tunisia

3 Instituto Politécnico de Coimbra, Rua da Misericórdia, Lagar dos Cortiços, S. Martinho do Bispo, 3045-093 Coimbra, Portugal

4 LAQV/REQUIMTE, Laboratory of Bromatology and Hydrology, Faculty of Pharmacy, University of Porto, Rua de Jorge Viterbo Ferreira, 228, 4050-313 Porto, Portugal

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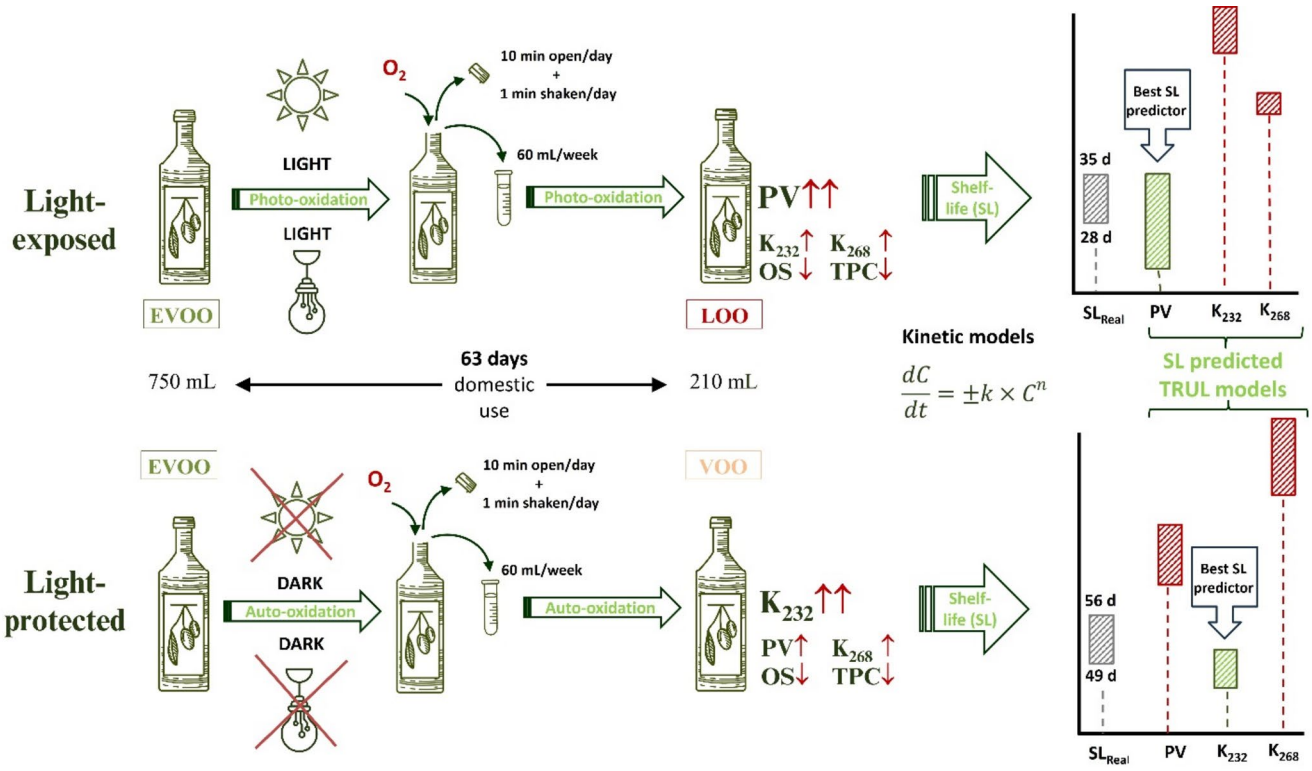
**Graphical Abstract**




**Keywords** Lipid photo-oxidation · Primary oxidation reactions · Kinetic degradation models · Reaction rate constants · TRUL models

## Introduction

Due to globalization, contemporary global food culture trends have emerged, yet without undermined consumer trust. Consumers still prefer traditional, locally sourced, organic, and slow food. As a result, traditional food producers face ongoing challenges in improving the safety and convenience of their products while emphasizing their nutritional benefits and health advantages and the need of ensuring their preservation throughout the commercialization chain. They strive to meet market demands and extend food products shelf-life (SL) while dealing with pressures from large retailers, open markets, and regulatory compliance [1].

Virgin olive oil is a key ingredient of the Mediterranean diet, highly appreciated for its sensory attributes and nutritional properties [2]. Besides its richness in unsaturated fatty acids, it contains bioactive compounds such as polyphenols, tocopherols, and carotenoids, which contribute to its antioxidant properties and health benefits [3]. Among these bioactive compounds, hydroxytyrosol-based phenolic compounds have recognised cardiovascular health benefits, supporting a health claim on the protection of blood lipids from oxidative stress when containing a minimum of 5 mg per 20 g of olive oil [4–6].

Based on specific legal thresholds for several physico-chemical and sensory parameters, olive oil can be commercially available as extra virgin (EVOO) or virgin olive oil (VOO), being classified as unsuitable for consumption (lampante olive oil– LOO) when not complying with these thresholds [7].

Even bottled, EVOO is naturally prone to oxidative degradation due to its high unsaturated fatty acid content, one of its main nutritional attributes. Two primary oxidative mechanisms are responsible for this degradation: photo-oxidation, occurring under light exposure, and auto-oxidation, which occurs both in the dark and under light. The presence of pro-oxidant factors as chlorophylls, free fatty acids, and trace metals, can accelerate oxidation [8, 9], while some minor components, as polyphenols, tocopherols, and carotenoids are known to counteract these oxidative processes [10]. Oxidation leads to the formation of undesirable volatile compounds, such as aldehydes and ketones, which contribute to sensory defects like rancidity [11, 12], at the expense of unsaturated fatty acids and antioxidants, reducing the oil's

nutritional value, and potentially disqualifying it from being labelled as EVOO or VOO, or even with specific health claims. The degradation of olive oil during storage has been extensively studied, under different conditions of temperature, light exposure, and packaging materials [13]. By monitoring the evolution of physicochemical parameters, bioactive compound concentration, which include minor compounds like phenolics, tocopherols and o-diphenols, as well as sensory characteristics, researchers have attempted to estimate olive oil's SL through empirical and kinetic models [2, 14–16]. For EVOO (or VOO), the term SL refers to the period during which the oil maintains its sensory attributes (e.g., perceived fruitiness, and absence of sensory defects) and physicochemical parameters (e.g., free acidity, FA; peroxide value, PV; and extinction coefficients at 232 and 268 nm, $K_{232}$ and $K_{268}$, respectively) within the regulatory limits under standard storage conditions [17, 18]. Additionally, the SL concept may include nutritional or health claims if stated on the oil's label. The SL concept may be further extended beyond commercial storage (i.e., prior to sale) to include the oil's SL during domestic use under typical household conditions. Two modelling approaches have been applied in the literature for predicting SL: time to reach the legal upper limit (TRUL) or time to reach the lower legal limit (TRLL), also referred to as the time to lose the health claim (TLHC) [2, 16]. However, the oxidative processes in unopened bottles stored in the dark differ markedly from those occurring during household use, where frequent bottle opening introduces oxygen and accelerates primary oxidation, with also frequent exposure to light, being this latter case the focus of the present study.

Indeed, only a few studies have focused on the effects of typical household consumption conditions on the oil's physicochemical and sensory characteristics, as well as its polyphenol content [18–20]. Although these studies used EVOOs with highly different physicochemical characteristics and varied in terms of storage conditions (20 and 37ºC; 28 to 120 days; and dark/light environments) and packaging materials (glass bottles, plated steel containers and bag-in-box multilayer pouches), they consistently reported a decline in oil quality over time, with some oils experiencing changes that affected their commercial classification in a short time [18, 19]. On the other hand, Klisović et al. [20] found that domestic use with storage under dark did not significantly affect the oil's commercial grade nor the polyphenol content related to the health claim, which remained above the EU-specified minimum during a one-month period.

However, none of these studies have developed a predictive strategy for estimating the SL of EVOO classification under typical household consumption/storage conditions. The present study aims to overcome this gap by specifically developing kinetic models to predict the SL of EVOO, from cv. Cobrançosa, in terms of both commercial quality (using TRUL models) and/or health claim status (using TRLL or TLHC model), of oils stored under light/dark conditions during household simulated consumption conditions. The findings of the present study may also contribute to informing consumers on the best storage conditions under domestic use as well as a possible "best-before date" for consumption after oil's bottle first opening [7, 21].

## Materials and methods

### Olive oil samples and simulated household consumption/storage conditions

Ten amber 750 mL glass bottles of cv. Cobrançosa EVOO from the same batch were obtained at Cooperativa de Olivicultores de Valpaços (Portugal). Although the fatty acids profile of the oils under study was not assessed, based on the literature data for olive oils industrially extracted from cv. Cobrançosa oil, typical relative abundances are expected, namely between 75 and 80% of monounsaturated fatty acids (with C18:1 the most abundant) and between 6 and 9% of polyunsaturated fatty acids (with C18:2 followed by C18:3, the most abundant ones) [22–25]. The bottles were stored for 9 weeks (63 days) in a laboratory at room temperature (18 ± 2ºC) under two distinct lighting conditions. Five bottles were stored in the darkness while the remaining were exposed to non-controlled day/night lights corresponding to the daily exposure to sunlight and artificial lights. Each day, all bottles were opened for 10 min and shaken for 1 min to allow oxygenation, simulating the frequent opening of bottles during domestic daily meal preparations. Additionally, weekly, 60 mL of oil was withdrawn from each bottle to mimic typical domestic use, which progressively increased the headspace within the bottles. The removed EVOO was subjected to physicochemical and sensory analyses as described in the following subsections.

### Olive oil quality parameters

During the 9-week time period, free acidity (FA), peroxide value (PV), specific UV absorbances at 232 nm ($K_{232}$) and 268 nm ($K_{268}$) parameters were monitored following EU official guidelines [26].

### Oxidative stability and total reducing capacity

Oxidative stability (OS) was measured in a Rancimat 743 equipment (Metrohm CH, Switzerland), at 120.0 ± 1.6°C, with an air supply of 20 L/h. Volatiles were collected in

water, whose conductivity (µS/cm) was continuously monitored, with the OS value (in h) corresponding to the inflection point of the conductivity curve.

The total reducing capacity (TRC) was quantified as the total phenolic content of the methanol-water extracted compounds based on the Folin-Ciocalteu reaction products measured spectrophotometric at765 nm (UV/Vis-1280 Shimadzu), as described by Pizarro et al. [27], quantified in milligrams of gallic acid equivalents (GAE) per kilogram of oil.

## Total content of hydroxytyrosol and tyrosol derivatives after acid hydrolysis

The quantification of total hydroxytyrosol and tyrosol was performed following acid hydrolysis based on the method described by Romero and Brenes [28], with modifications. A direct hydrolysis with 2 mol/L hydrochloric acid in methanol/water (80:20, v/v) was conducted in the dark, at 25°C, for 6 h with intermittent vortex agitation. Separation was achieved on a Gemini Nx C18 column (5 µm particle size, 110 Å pore size, 150 mm × 4.60 mm, Phenomenex) at 35 °C, under isocratic elution with acetonitrile/water (14:86 v/v, 2 mL/min) in a Jasco HPLC system (Japan) with a DAD detector (MD-4010). Calibration curves for hydroxytyrosol and tyrosol were established under the same hydrolysis conditions, all using syringic acid as internal standard, extracted at 280 nm. Results are expressed as mg/kg of oil, after the application of the correction factor of 2.2 for hydroxytyrosol and the correction factor of 2.5 for tyrosol, to estimate their original molecular forms [29].

## Descriptive sensory analysis

Sensory analysis was performed by the sensory panel of Escola Superior Agrária of the Instituto Politécnico de Bragança according to the International Olive Council [30] with some modifications [31]. The sensory panel consists of eight trained panellists (five men and three women) aged between 25 and 50 years. The test sheet used for the descriptive sensory analysis included olfactory and taste sensations, which were assessed using an unstructured intensity scale ranging from 0 (no sensation perceived) to 10 (maximum intensity of sensation perceived). Olfactory sensations included ripe or green fruity notes, fruity and herbaceous aromas, while taste sensations included ripe or green fruity taste, sweetness, bitterness, pungency, fruity and herbaceous attributes. Additionally, the harmony was assessed as olfactory or gustatory sensations contributing to an overall pleasant sensation. Harmony represents an overall perception that combines all sensations experienced by the panellist, reflecting the equilibrium among these perceptions. Higher scores are typically assigned when multiple sensations are detected without any single one being overly dominant [31]. Furthermore, two qualitative gustatory-retronasal sensations, namely complexity and persistence, were also evaluated. Complexity was assessed based on the combination of different positive sensations perceived in each olive oil, increasing when a greater variety of sensations is present and decreasing when fewer are detected [31]. As for persistence, its intensity refers to the duration for which the retronasal sensation persists after the olive oil is no longer in the mouth, with longer durations indicating higher persistence [30, 31]. All samples were analysed simultaneously, with the samples collected weekly preserved at 4ºC.

## Shelf-life kinetic models

SL kinetic models rely on estimating reaction rates ($k$) for key physicochemical parameters, assuming a specific reaction order ($n \geq 0$). For that a relationship between the content/level of a specific parameter of interest (C) and time (t) can be established.

$$\frac{dC}{dt} = \pm\ k \times\ C^n \tag{1}$$

The positive or negative sign (±) in the equation reflects the increasing or decreasing trend of the parameter over time, respectively. The integration of Eq. (1), from the initial storage conditions ($t=0$ and $C=C_0$) and the current conditions (t and C) will result in different mathematical models depending on the assumed reaction order.

Zero-order reaction ($n=0$):

$$\frac{dC}{dt} = \pm\ k \rightarrow\ integration:\ C - C_0 = \pm\ k \times\ t \tag{2}$$

First-order reaction ($n=1$):

$$\frac{dC}{dt} = \pm\ k \times\ C \rightarrow\ integration:\ ln\left(\frac{C}{C_0}\right) = \pm\ k \times\ t \tag{3}$$

Other-order reaction ($n \neq \{0,1\}$):

$$\frac{dC}{dt} = \pm\ k \times\ C^n \rightarrow\ integration:\ \frac{C^{-n+1}}{-n+1} - \frac{C_0^{-n+1}}{-n+1} = \pm\ k \times\ t \tag{4}$$

By employing experimental data (t, C) and using Eq. (2) to (4), the kinetic reaction rates at a constant temperature can be estimated by linear regression analysis, with k values equal to the linear slopes. According to Eq. (2) to (4), if the first term in each equation is taken as the ordinate (y-axis) and the second term as the abscissa (x-axis) for the regression analysis, theoretically the line's intercept should

be zero. Therefore, the regression analysis should be conducted with the intercept constrained to zero, which may yield slightly different values compared to an unconstrained analysis. The quality of the fit can be evaluated using the regression's correlation coefficient (R values).

Two distinct types of kinetic models were used to predict the SL [2, 16]: the time to reach the legal upper limit (TRUL), for parameters that increase with the storage time (e.g., FA, PV, $K_{232}$ or $K_{268}$), and the time to reach the legal lower limit (TRLL), for parameters whose levels decrease with the storage time (e.g., polyphenols content related to the health claim; also referred as the time to lose the health claim, TLHC).

The SL was determined as follows.

Zero-order reaction ($n=0$):

$$SL = \frac{C_{legal\ limit} - C_0}{\pm\ k} \tag{5}$$

First-order reaction ($n=1$):

$$SL = \frac{ln\left(\frac{C_{legal\ limit}}{C_0}\right)}{\pm\ k} \tag{6}$$

Other-order reaction ($n \neq \{0,1\}$)

$$SL = \frac{\frac{C_{legal\ limit}^{-n+1}}{-n+1} - \frac{C_0^{-n+1}}{-n+1}}{\pm\ k} \tag{7}$$

### Statistical analysis

The significance of the studied simulated domestic storage conditions, on the physicochemical, sensory and health claim data was statistically evaluated. For the storage time-period, one-way analysis of variance (ANOVA) was applied, followed by the Tukey's multicomparison test when a significant statistical effect was found. For the lighting conditions (i.e., darkness *versus* light exposition) a t-Student's test was applied. Data are reported as the mean ± standard deviation (SD) of triplicate analysis of five independent olive oil bottles. Linear Discriminant Analysis (LDA) was also employed to assess if the domestic use time-periods (ranging from 0 to 63 days) could be identified for the studied light/dark conditions. Optimal parameter subsets were identified by the meta-heuristic simulated annealing (SA) algorithm. The classification performance of each model was assessed using sensitivity metrics for the training dataset, leave-one-out cross-validation (leave-one-out-CV), and repeated K-fold-CV (4 folds with 10 random repetitions). To further evaluate classification performance, 2-D plots of the first two discriminant functions (DFs) derived from the original grouped data were used. Confidence ellipses were constructed based on posterior probabilities, calculated via Bayes' theorem for each defined class. The kinetic reaction rates and respective standard errors, of zero-, first- and second-order models, were estimated by simple linear regression analysis. All statistical analyses were performed using RStudio (version 3.6.2), with the significance level set equal to 5%.

## Results and discussion

### Physicochemical quality parameters

Under simulated household conditions, both storage time (0 to 63 days) and lighting conditions (dark *versus* light exposure) had, in general, statistically significant effects (P-value < 0.05) on oxidation parameters, namely PV, $K_{232}$, and $K_{268}$ as shown in Table 1. Throughout the 9-week study period, the PV, which is influenced by the formation of primary oxidation products like hydroperoxides and peroxides from unsaturated fatty acids, showed the greatest increase. Oils exposed to light exhibited a higher rise (+435%), from 10.6 to 56.7 meq. $O_2$/kg, in comparison to those stored in darkness (+80%), which increased from 10.6 to 19.1 meq. $O_2$/kg, highlighting the negative effects of photo-oxidation in combination with oxygen availability. The oils stored in darkness retained their EVOO classification during the entire assay, while those exposed to light exceeded the legal EU limit of 20 meq. $O_2$/kg of oil (between days 28 and 35) [26], disqualified to the LOO grade, making them unsuitable for consumption. Simultaneously, the $K_{232}$ values, indicative of conjugated dienes derived from polyunsaturated fatty acids primary oxidation, favoured by oxygen availability, experienced a similar rose from 1.81 (t = 0 days) to 2.46 and 2.59 in light-exposed and dark-stored oils (t = 63 days), respectively (+36% and +43%, respectively). The observed maximum values remained below the EU limits for EVOO (2.50) classification [26] up to week seven (day 49) and for VOO (2.60) classification [26] until the end of the study. The faster rise of PV compared to $K_{232}$ is a direct consequence of the olive oil richness in monounsaturated fatty acids, not contributing directly to the formation of conjugated dienes, typical of polyunsaturated fatty acids. Similarly, $K_{268}$ values, measuring conjugated fatty acid trienes, showed minimal variation, from 0.16 to 0.18 in oils stored in darkness and to 0.22 in oils exposed to light, remaining at or below the EU's legal limit for EVOO (0.22) [26], again as expected for low polyunsaturated oils and storage conditions (reduced time and low temperatures). In general, the observed increasing trends of PV and $K_{232}$ over time align

**Table 1** Mean (±standard deviation) values for free acidity (FA), peroxide value (PV), extinction coefficients at 232 and 268 nm ($K_{232}$ and $K_{268}$), oxidative stability (OS), total reducing capacity (TRC), and total content of hydroxytyrosol and tyrosol derivatives after acid hydrolysis (EFSA health claim) of cv. Cobrançosa EVOO over 9 weeks (a 63-day time-period) of simulated household consumption conditions (daily opening and shaken with oil weekly withdrawn) stored in darkness (light-protected oils) or exposed to natural and artificial light (light-exposed oils) at ambient temperature (18 ± 2ºC)

| Parameters | Storage conditions | Storage time | | | | | | | | | | P-value[#] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0 days | 7 days | 14 days | 21 days | 28 days | 35 days | 42 days | 49 days | 56 days | 63 days | |
| FA (%) | Light | 0.23 ± 0.00$^{b}$ | 0.23 ± 0.01$^{abA}$ | 0.23 ± 0.00$^{bA}$ | 0.23 ± 0.01$^{abA}$ | 0.23 ± 0.00$^{bA}$ | 0.23 ± 0.01$^{abA}$ | 0.25 ± 0.02$^{aA}$ | 0.23 ± 0.01$^{abA}$ | 0.24 ± 0.02$^{abA}$ | 0.23 ± 0.01$^{abA}$ | 0.0259 |
| | Dark | 0.23 ± 0.00$^{a}$ | 0.24 ± 0.02$^{aA}$ | 0.23 ± 0.01$^{aA}$ | 0.23 ± 0.00$^{aA}$ | 0.23 ± 0.00$^{aA}$ | 0.23 ± 0.01$^{aA}$ | 0.24 ± 0.02$^{aA}$ | 0.23 ± 0.00$^{aA}$ | 0.23 ± 0.00$^{aA}$ | 0.23 ± 0.01$^{aA}$ | 0.2690 |
| | P-value[§] | – | 0.2400 | 0.3450 | 0.3870 | 0.2970 | 0.9980 | 0.3460 | 0.3480 | 0.1420 | 0.9999 | – |
| PV (meq. $O_2$/kg of oil) | Light | 10.6 ± 0.2$^{h}$ | 14.6 ± 0.4$^{gA}$ | 14.6 ± 0.1$^{gA}$ | 14.8 ± 0.2$^{gA}$ | 17.4 ± 0.2$^{fA}$ | 21.5 ± 0.4$^{eA}$ | 26.9 ± 0.3$^{dA}$ | 32.1 ± 2.2$^{cA}$ | 33.8 ± 0.31$^{bA}$ | 56.7 ± 1.0$^{aA}$ | < 0.0001 |
| | Dark | 10.6 ± 0.2$^{d}$ | 10.8 ± 0.0$^{dB}$ | 12.6 ± 0.3$^{cB}$ | 12.7 ± 0.2$^{cB}$ | 13.1 ± 0.4$^{cB}$ | 13.6 ± 0.2$^{cB}$ | 16.5 ± 0.2$^{bB}$ | 18.1 ± 1.4$^{aB}$ | 18.8 ± 0.3$^{aB}$ | 19.1 ± 0.4$^{aB}$ | < 0.0001 |
| | P-value[§] | – | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | – |
| $K_{232}$ | Light | 1.81 ± 0.02$^{f}$ | 1.93 ± 0.04$^{efA}$ | 2.01 ± 0.07$^{deB}$ | 2.06 ± 0.15$^{deB}$ | 2.08 ± 0.06$^{cdeB}$ | 2.11 ± 0.05$^{cdB}$ | 2.21 ± 0.03$^{bcB}$ | 2.29 ± 0.05$^{bB}$ | 2.36 ± 0.09$^{abB}$ | 2.46 ± 0.04$^{aB}$ | < 0.0001 |
| | Dark | 1.81 ± 0.02$^{d}$ | 2.02 ± 0.11$^{dA}$ | 2.25 ± 0.03$^{cA}$ | 2.28 ± 0.12$^{cA}$ | 2.35 ± 0.05$^{cA}$ | 2.39 ± 0.05$^{cA}$ | 2.43 ± 0.03$^{bA}$ | 2.49 ± 0.03$^{aA}$ | 2.54 ± 0.03$^{abA}$ | 2.59 ± 0.03$^{aA}$ | < 0.0001 |
| | P-value[§] | – | 0.1450 | < 0.0001 | 0.0342 | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | 0.0025 | 0.0004 | – |
| $K_{268}$ | Light | 0.16 ± 0.02$^{e}$ | 0.16 ± 0.01$^{eA}$ | 0.17 ± 0.01$^{deA}$ | 0.18 ± 0.01$^{cdA}$ | 0.19 ± 0.00$^{cA}$ | 0.19 ± 0.01$^{cA}$ | 0.20 ± 0.01$^{bcA}$ | 0.21 ± 0.00$^{abA}$ | 0.22 ± 0.00$^{aA}$ | 0.22 ± 0.00$^{aA}$ | < 0.0001 |
| | Dark | 0.16 ± 0.02$^{b}$ | 0.16 ± 0.01$^{bA}$ | 0.17 ± 0.00$^{abA}$ | 0.17 ± 0.01$^{abB}$ | 0.18 ± 0.00$^{aB}$ | 0.17 ± 0.01$^{abB}$ | 0.17 ± 0.00$^{abB}$ | 0.18 ± 0.00$^{aB}$ | 0.18 ± 0.01$^{aB}$ | 0.18 ± 0.01$^{aB}$ | < 0.0001 |
| | P-value[§] | – | 0.6210 | 0.4290 | 0.0274 | 0.0002 | 0.0002 | < 0.0001 | < 0.0001 | < 0.0001 | < 0.0001 | – |
| OS (h) | Light | 9.3 ± 0.7aA | 9.3 ± 0.2aA | 9.0 ± 0.2abA | 9.1 ± 0.2abA | 8.5 ± 0.1bA | 7.9 ± 0.19cB | 7.4 ± 0.3cB | 6.3 ± 0.1dB | 6.0 ± 0.2dB | 9.31 ± 0.73aA | < 0.0001 |
| | Dark | 9.3 ± 0.3abA | 9.0 ± 0.2abcdA | 8.9 ± 0.2abcdA | 9.4 ± 0.3aA | 8.7 ± 0.6bcdA | 9.0 ± 0.4abcdA | 8.6 ± 0.2cdA | 7.9 ± 0.2eA | 8.5 ± 0.2deA | 9.3 ± 0.3abA | < 0.0001 |
| | P-value[§] | – | 0.9650 | 0.1080 | 0.3650 | 0.0939 | 0.5440 | 0.0004 | < 0.0001 | < 0.0001 | < 0.0001 | – |
| TRC (mg GAE/kg oil) | Light | 556 ± 6a | 566 ± 11aA | 562 ± 26aA | 464 ± 16bB | 455 ± 11bB | 432 ± 45bB | 424 ± 29bA | 465 ± 19bB | 464 ± 101bA | 462 ± 24bA | < 0.0001 |
| | Dark | 556 ± 6a | 517 ± 16aB | 531 ± 47aA | 502 ± 6aA | 501 ± 20aA | 516 ± 19aA | 513 ± 118aA | 517 ± 29aA | 501 ± 18aA | 472 ± 26aA | 0.2970 |
| | P-value[§] | – | 0.0006 | 0.2310 | 0.0010 | 0.0019 | 0.0048 | 0.1390 | 0.0101 | 0.4430 | 0.5290 | ---- |
| Hydroxy-tyro-sol+tyrosol derivatives (mg/20 g oil) | Light | 12.5 ± 0.4$^{a}$ | 12.9 ± 0.5$^{aA}$ | 13.1 ± 0.6$^{aA}$ | 12.8 ± 0.5$^{aA}$ | 12.6 ± 0.2$^{aA}$ | 12.4 ± 0.4$^{aA}$ | 12.1 ± 0.6$^{aA}$ | 12.4 ± 0.9$^{aA}$ | 12.2 ± 0.5$^{aA}$ | 12.4 ± 0.6$^{aA}$ | 0.1380 |
| | Dark | 12.5 ± 0.4$^{a}$ | 12.8 ± 0.2$^{aA}$ | 12.7 ± 0.2$^{aA}$ | 12.4 ± 0.3$^{aA}$ | 12.2 ± 0.4$^{aB}$ | 12.1 ± 0.4$^{aA}$ | 12.1 ± 0.7$^{aA}$ | 12.6 ± 0.3$^{aA}$ | 12.3 ± 0.4$^{aA}$ | 12.2 ± 0.4$^{aA}$ | 0.0532 |
| | P-value | – | 0.5730 | 0.1310 | 0.1970 | 0.0478 | 0.3370 | 0.9400 | 0.7160 | 0.8040 | 0.5720 | – |

[#]One-way ANOVA: different lowercase letters at each household lighting storage condition and for each parameter indicate a statistically significant effect (P-value < 0.05) of the storage time-period (from 0 to 63 days), according to the multicomparison Tukey's test

[§]t-Student's test: different uppercase letters at each household storage time-period and for each parameter indicate a statistically significant effect (P-value < 0.05) of the two studied lighting conditions (dark versus light exposure)

with those previously reported by other researchers, who also studied EVOO degradation during mimicked household consumption conditions [18–20].

It should be noted that the previous studies used EVOOs with different initial oxidation levels and with varied storage conditions (e.g., 20 to 37ºC; 28 to 120 days; dark or light environments) and packaging materials (glass bottles, plated steel containers and bag-in-box multilayer pouches), which may explain some of the differences in terms of oxidation extent and final quality grade of the oils. For example, Rodrigues et al. [18] found that for 28 days, the EVOO quality grade of light-exposed EVOO stored in amber glass bottles that were left open or closed after each simulated use diminished to LOO due to $K_{232}$ increase and not due to PV that, despite doubling during the time-period of the assay, was still below the legal limit. The oils used by these authors had low initial PV values (4.6 to 5.8) but relatively high $K_{232}$ initial values (between 1.94 and 2.29, close to the EU legal limits for EVOO or VOO classification). In the study by Klisović et al. [20], the degradation of EVOO from two different cultivars was monitored over 28 days also simulating typical domestic use with oils stored in the dark at 20 ± 2ºC. Results showed no significant changes in $K_{268}$ values, while $K_{232}$ and PV increased slightly over time but remained within the EU's legal limits for EVOO classification, being hypothesized by those researchers that the degradation degree could be predominantly attributed to oil ageing and not to domestic use. It should be noted that, in the present study, by day 28 of simulated domestic use, both the light-protected and light-exposed oils maintained their EVOO classification despite showing higher oxidation levels. Lolis et al. [19] also observed an increasing level of the extinction coefficients and the PV over time. However, a more detailed comparison may not be fair since the household consumption/storage conditions differ significantly when compared to the present study as well as those of Rodrigues et al. [18] and Klisović et al. [20]. Indeed, Lolis et al. [19] monitored the oil's changes during a four-month period under household and abuse storage temperature (22 and 37ºC) conditions, being the oils stored in plated steel containers and bag-in-box multilayer pouches, with different oil removal scheduling (every 20 days). Concerning lipolysis, the FA levels remained relatively stable (Table 1), ranging from 0.23 to 0.25%, well below the legal limit of 0.8% for EVOO classification, suggests that no accelerated triglyceride hydrolysis, the primary driver of FA increase in olive oils under the studied conditions.

## Oxidative stability and total reducing capacity

The evolution of the oxidative stability with time and antioxidant capacity are presented in Table 1. Storage time influenced OS and TRC (P-value < 0.05) after the 21st day, particularly in light-exposed oils, with higher losses in OS (−34%) and TRC (−24%) compared to light-protected ones (−7 and −15%, respectively) already on the 28th day and until the 56th day, and with an inconsistent increase from that date to the end of the study. The more pronounced decrease observed in light-exposed oils is consistent with the oxidation status observed in the quality parameters and with the consequent reduction of phenolic-like antioxidants. The observed TRC decrease may be partially responsible for the greater oil's quality degradation since, as pointed out in the literature, olive phenols can contribute to extend the SL of stored food products namely by retarding fats deterioration [32]. However, Rodrigues et al. [18] observed no meaningful variation in OS during the domestic use period of light-exposed oils, a finding likely influenced by the shorter study duration (28 days). As to the TRC, Lolis et al. [19] reported a similar decreasing trend over a more extended period of domestic use (120 days). A similar, though less pronounced, decrease in TRC was also found, over a shorter time-period of 28 days, by Rodrigues et al. [18] reporting a 16% reduction in light-exposed oils, and by Klisović et al. [20], with reductions ranging from 12 to 18% in light-protected oils, varying according to the olive cultivar.

## Total content of hydroxytyrosol and tyrosol derivatives and related health claim

The results (Table 1) showed that the simulated household consumption/storage conditions had a minor influence on the content of bioactive phenols related to the EFSA health claim (12.5–13.1 mg/20 g of oil, P-value > 0.05), remaining significantly above the minimum amount required by the claim (5 $mg_{hydroxytyrosol+tyrosol\ derivatives}$/20 g of oil). Similarly, Klisović et al. [20] also reported the preservation of the initial total content of bioactive phenols related to the EFSA health claim, even knowing that a different analytical method was used, after acid hydrolysis in the present study and based on the sum of individual phenols (hydroxytyrosol, tyrosol, hydroxytyrosol acetate, 3,4-DHPEA-EDA, p-HPEA-EDA, oleuropein aglycones, and ligstroside aglycones) by Klisović et al. [20]. The specific preservation of these phenolic compounds sustains a hypothesis that although they are recognized antioxidants that contribute to the preservation of blood lipids from oxidative stress, as detailed in the health claim, their capacity to counteract the oxidation phenomena occurring in the olive oil might be reduced, with other more lipidic antioxidants playing more relevant parts, as might be expected from tocopherols or even sterols.

## Sensory descriptive profile: intensity of olfactory and gustatory sensations

Tables 2 and 3 present the mean intensities of the perceived olfactory and gustatory sensations. Across all samples, the panellists identified the same nine positive olfactory and twelve positive gustatory sensations. The intensity of "fresh" olfactory and gustatory sensations (e.g., apple, tomato, tomato leaves, cabbage and fresh grass) in both light-protected and light-exposed oils progressively declined with the domestic storage time, typically disappearing between days 35 and 42, except for apple sensation, which persisted throughout the 63-day study. Overall, the decline in sensation intensities was faster in oils exposed to light than in those stored in darkness. On the contrary, the intensity of "dry" olfactory and gustatory sensations (e.g., dry grass) increased over the course of the study. Regarding the two basic tastes (bitterness and sweetness) and the trigeminal sensation (pungency), a general increase in sweetness was observed, particularly in light-protected oils, while both bitterness and pungency decreased, but at a faster rate in light-exposed oils. These observations align with previous literature findings regarding the domestic storage of EVOO in glass bottles over a 28-day period, under both light exposure [18] and darkness [20].

Remarkably, an intense rancid olfactory sensation was detected by the sensory panel in light-exposed oils by day 56 (mean intensity of 3.60), which almost doubled by day 63 (mean intensity of 6.10), leading to EVOO declassification to LOO. This outcome is in-line with the high mean PV found in light-exposed oils, at day 56 (33.8 meq. $O_2$/kg oil) and at day 63 (56.7 meq. $O_2$/kg oil), suggesting substantial oxidative degradation, likely due to the formation of several volatile secondary oxidation products (such as aldehydes and ketones), which are known to contribute to the appearance of rancid defect [11, 12]. Similar findings were previously reported by Rodrigues et al. [18] with rancidity notes in light-exposed EVOO after 28 days of domestic use, though at lower intensities (from 1.10 to 1.55). In contrast, no sensory defects were noted in light-protected oils in this study, consistent with findings reported by Klisović et al. [20]. Finally, regarding overall sensory perceptions, both light-exposed and light-protected oils exhibited olfactory and gustatory harmony intensities ranging from 7.4 to 8.4, which slightly declined over the 63-day period of domestic use. Similarly, the intensities of the two overall qualitative gustatory-retronasal sensations, i.e., complexity and persistence, also diminished with time, with a more pronounced reduction observed in the light-exposed oils (final values of 3.7). These results align with the findings of Rodrigues et al. [18], who reported a comparable decline in these attributes over a 28-day period.

## Discrimination of domestic use time-period based on physicochemical data and sensory profiles

Independent LDA models were constructed for oils stored under light-exposed and light-protected conditions to access the discriminative potential of the physicochemical and sensory parameters evaluated. For oils exposed to light, a multivariate classification model was developed using a subset of five parameters ($K_{268}$, and the intensities of olfactory fruity green, as well as of gustatory tomato, tomato branches, and cabbage). This model achieved a 98% correct classification rate on both the training data (Fig. 1A) and leave-one-out-CV. The only misclassification occurred when an oil sample with 42 days of domestic use/storage was incorrectly classified as being a sample with 49 days of domestic use/storage. Repeated K-fold-CV yielded an accuracy of 98 ± 5%, with a higher number of misclassifications between the same two groups of samples. For light-protected oils, a supervised LDA model was developed based on a more extensive set of 13 parameters (PV, $K_{268}$, TRC, and the intensities of olfactory fruity green, tomato, and dry herbs, as well as gustatory fruity ripe, fruity green, bitter, pungent, dry fruits, tomato branches, and dry herbs). This model allowed classification accuracies of 100% for training data (Fig. 1B), 98% for leave-one-out-CV, where two oils with 56 days of domestic use/storage were misclassified as oils with only 49 days of use/storage, and 96 ± 5% for repeated K-fold-CV, with misclassifications between 49-day and 56-day samples and more rarely classifying 63-day samples as 56-day. These findings confirm that the simulated household storage conditions significantly influenced the temporal evolution of physicochemical properties and sensory intensities, enabling the identification of specific biomarkers for time-period discrimination. Moreover, the analysis demonstrated that light exposure led to more extensive degradation and pronounced changes in the measured parameters. This was evidenced by the reduced number of parameters (5 versus 13) required for accurate temporal classification in light-exposed compared to light-protected oils.

## Kinetic models, degradation rate constants and predicted shelf-life

Predicting the SL of an EVOO during its domestic use is of utmost importance for consumers and producers. However, although predictive kinetic models have been proposed in the literature for estimating the SL of olive oils, in terms of preservation of their commercial grade in unopened containers under different storage conditions [15], to the authors' best knowledge no study addressed the SL prediction under typical household consumption and storage conditions.

**Table 2** Mean (±standard deviation) intensities of olfactory sensations perceived in cv. Cobrançosa EVOO by a trained panel (0: not perceived to 10: maximum perceived intensity) over a 63-day time-period of simulated household consumption conditions (daily opening and shaken with oil weekly withdrawn) stored in darkness (light-protected oils) or exposed to natural and artificial light (light-exposed oils) at ambient temperature (18±2ºC)

| Olfactory sensation | Storage conditions | Storage time | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0 days | 7 days | 14 days | 21 days | 28 days | 35 days | 42 days | 49 days | 56 days | 63 days | *P*-value[#] |
| Positive sensations | | | | | | | | | | | | |
| Ripe fruity | Light | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | 5.6±1.5aA | 6.1±1.0aA | N.E. | N.E. | 0.5420 |
| | Dark | | N.D. | N.D. | N.D. | N.D. | N.D. | 3.9±1.3bA | 5.1±0.4abA | 5.1±0.4ab | 5.4±0.2a | 0.0262 |
| | P-value[§] | – | – | – | – | – | – | 0.0999 | 0.0711 | – | – | – |
| Green fruity | Light | 6.8±0.6a | 5.8±0.2bB | 5.6±0.4bA | 5.6±0.5bA | 4.6±0.9cA | 3.3±0.3dA | N.D. | N.D. | N.E. | N.E. | <0.0001 |
| | Dark | | 6.4±0.3aA | 5.7±0.3bA | 4.6±0.2cB | 2.6±0.3dB | 1.5±0.2eB | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value§ | – | 0.0039 | 0.5710 | 0.0012 | 0.0014 | <0.0001 | – | – | – | – | – |
| Apple | Light | 5.9±0.4a | 5.3±0.2bA | 4.5±0.3cA | 4.3±0.2cA | 3.3±0.2dA | 3.4±0.4dA | 2.4±0.4eA | 2.6±0.3eA | N.E. | N.E. | <0.0001 |
| | Dark | | 5.2±0.5bA | 4.3±0.3cA | 3.6±0.3cdB | 3.4±0.4dA | 2.7±0.2eB | 2.6±0.1eA | 2.5±0.2eA | 2.4±0.3e | 2.6±0.3e | <0.0001 |
| | P-value§ | – | 0.7910 | 0.3870 | 0.0062 | 0.4840 | 0.0092 | 0.3550 | 0.8560 | – | – | – |
| Tomato | Light | 6.7±0.7a | 6.1±0.1abA | 5.6±0.2bA | 5.5±0.3bA | 4.5±0.2cA | 4.3±0.4cA | N.D. | N.D. | N.E. | N.E. | <0.0001 |
| | Dark | | 6.3±0.4abA | 5.6±0.2bA | 4.6±0.3cB | 3.2±0.3dB | 2.6±0.3dB | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.1640 | 0.8160 | 0.0009 | <0.0001 | <0.0001 | – | – | – | – | – |
| Dry fruits | Light | 2.8±0.2d | 3.9±0.5cA | 4.5±0.3bcA | 4.5±0.5bcA | 4.7±0.4bcA | 5.1±0.6bA | 6.2±0.5aA | 6.3±0.4aA | N.E. | N.E. | <0.0001 |
| | Dark | 2.8±0.2ef | 2.7±0.4fB | 3.8±0.2cdB | 3.9±0.2cdB | 3.4±0.3deB | 3.9±0.5cdB | 4.3±0.5abcB | 4.0±0.3bcdB | 4.6±0.3ab | 4.9±0.4a | <0.0001 |
| | P-value[§] | – | 0.0031 | 0.0031 | 0.0310 | 0.0004 | 0.0109 | 0.0003 | <0.0001 | – | – | – |
| Tomato leaves | Light | 5.3±0.3a | 4.7±0.8abA | 4.4±0.3bA | 4.1±0.2bcA | 3.3±0.2cA | N.D. | N.D. | N.D. | N.E. | N.E. | <0.0001 |
| | Dark | | 4.7±0.3bA | 3.9±0.1cB | 3.8±0.2cB | 2.8±0.4dB | 2.5±0.2d | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.9999 | 0.0068 | 0.039 | 0.0441 | – | – | – | – | – | – |
| Cabbage | Light | 6.3±0.6a | 5.4±0.4bA | 5.3±0.4bA | 4.4±0.3cA | 3.4±0.2dA | N.D. | N.D. | N.D. | N.E. | N.E. | <0.0001 |
| | Dark | | 5.5±0.4bA | 4.7±0.2cB | 4.1±0.4cdA | 3.5±0.2dA | 2.2±0.3e | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.7760 | 0.0089 | 0.1730 | 0.5330 | – | – | – | – | – | – |
| Fresh grass | Light | 5.1±0.2a | 4.5±0.4abA | 3.6±0.4bcA | 3.5±0.7cA | 3.3±0.5cA | 2.9±0.3cA | N.D. | N.D. | N.E. | N.E. | <0.0001 |
| | Dark | | 4.1±0.4bA | 3.6±0.1bcA | 3.3±0.5cA | 2.4±0.3dB | 2.3±0.4dB | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.1120 | 0.9600 | 0.6600 | 0.0087 | 0.0185 | – | – | – | – | – |
| Harmony | Light | 8.4±0.2a | 8.3±0.3abA | 8.3±0.2abA | 8.0±0.4abcA | 8.4±0.1abA | 7.8±0.2bcdB | 7.5±0.4cdA | 7.4±0.2dA | N.E. | N.E. | <0.0001 |
| | Dark | | 8.2±0.2abcA | 8.1±0.2abcA | 7.8±0.1cA | 8.5±0.2aA | 8.2±0.1abcA | 7.8±0.4bcA | 7.7±0.5cA | 7.8±0.3c | 7.8±0.4bc | 0.0002 |
| | P-value§ | – | 0.2510 | 0.2110 | 0.3880 | 0.2680 | 0.0029 | 0.2220 | 0.2480 | – | – | – |
| Negative sensations | | | | | | | | | | | | |
| Rancid | Light | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | 3.6±0.7b | 6.1±0.5a | 0.0008 |
| | Dark | | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | – |
| | P-value[§] | – | – | – | – | – | – | – | – | – | – | – |

N.D.: not detected; N.E.: not evaluated since an intense olfactory defect was perceived (intensity > 3.50)

[#] One-way ANOVA: different lowercase letters at each household lighting storage condition and for each parameter indicate a statistically significant effect (P-value < 0.05) of the storage time-period (from 0 to 63 days), according to the multicomparison Tukey's test

[§] t-Student's test: different uppercase letters at each household storage time-period and for each parameter indicate a statistically significant effect (P-value < 0.05) of the two studied lighting conditions (dark versus light exposure)

The studies on the commercial storage of unopened containers have proposed zero-, first, and second-order kinetic models for predicting the SL, with the latter being less commonly applied [15, 16, 33–35]. Accordingly, in this study, zero-, first-, and second-order kinetic models were developed to estimate the duration over which the oil retained the legal standards of EVOO commercial grade under typical domestic use conditions by applying TRUL models. Of the four monitored quality parameters, FA was disregarded due to its stability over the study period. Likewise, because the total content of hydroxytyrosol and tyrosol derivatives remained almost unchanged, it was not possible to develop a TRLL model to assess the SL of the related health claim. Knowing that sensory analysis is highly relevant to the EVOO classification but also that its evaluation is expensive and of reduced availability, TRUL models (zero-, first-, and second-order) were only established for the oxidation parameters (PV, $K_{232}$, and $K_{268}$). The reaction rate constants ($k$) were determined through linear regression (Eq. (2) to (4)). The fit quality (correlation coefficients, R) and $k$ values (± standard error) are shown in Table 4. The predicted SL (± standard deviation) calculated using Eq. (5) to (7) are also listed in Table 4.

Based on the R values, the zero-, first-, and second-order kinetic models demonstrated a satisfactory fit quality ($0.942 \leq R \leq 0.997$) for describing the three quality parameters over time for light-exposed and light-protected oils. For PV and $K_{268}$, the calculated $k$ values were generally more than 3-times higher in oils exposed to light compared to those protected from light, demonstrating that light exposure significantly accelerated oxidation reactions, and so, that photo-oxidation rates exceeded auto-oxidation rates. For $K_{232}$, the $k$ values were slightly higher in light-protected oils compared to light-exposed oils, though they were of the same order of magnitude.

Regarding SL predictions based on TRUL models, the results (Table 4) indicated that, in light-exposed oils, PV led to the lowest average SL predictions ($20 \leq SL \leq 35$ days), followed by $K_{268}$ ($56 \leq SL \leq 60$ days) and $K_{232}$ ($63 \leq SL \leq 73$ days). Experimentally, the SL for light-exposed oils was established between 28 and 35 days, as PV increased from 17.4 to 21.5 meq. $O_2$/kg oil over this period, exceeding the maximum legal limit for EVOO classification before day 35, decreasing the oil classification to LOO. In accordance, PV was identified as the most appropriate SL indicator under the simulated household consumption and storage conditions.

Additionally, a detailed analysis showed that for PV, the zero- and first-order models tended to underestimate the SL of light-exposed oils, while the second-order model proved to be the most accurate, predicting an SL of 35 ± 2 days, consistent with the experimentally determined SL range. For oils protected from light exposure, the experimental SL of EVOO was determined to be between 49 and 56 days. This was observed as the $K_{232}$ value exceeded the regulatory threshold for EVOO classification (2.50) by day 56 under the simulated domestic conditions, resulting in a downgrade to VOO, which remained until the study's conclusion on day 63. The SL for light-protected oils was approximately twice that of light-exposed oils, indicating a significant mitigating effect of darkness on oil oxidation and underscoring the negative influence of photo-oxidation. TRUL models based on zero-, first-, and second-order kinetics applied to $K_{268}$ data (Table 4) considerably overestimated the SL of light-protected EVOO, with average predictions ranging between 135 and 171 days. By contrast, TRUL models based on PV, while still overestimating the experimental SL, provided lower SL predictions, with average values ranging from 62 to 71.Contrary, $K_{232}$ proved to be the most realistic indicator of SL for oils stored in darkness under the studied simulated household conditions, predicting a SL of approximately 48–49 days, regardless of the order of the TRUL model. Nonetheless, the zero-order model provided a predicted SL of 49 ± 4 days, aligning most closely with the observed experimental range of 49–56 days.

Although the kinetic models were developed using data for EVOO of cv. Cobrançosa, the models' validity as well as of the corresponding estimated $k$ values (Table 4), were further assessed using the time-evolution data of PV, $K_{232}$, and $K_{268}$ reported by Rodrigues et al. [18] for light-exposed cv. Arbequina oils and by Klisović et al. [20] for light-protected cvs. Istarska Bjelica and Buža oils, under simulated household conditions at ambient temperatures over a 28-day period. Initial data from those two studies, in combination with the $k$ values estimated in the present study for the two different lighting conditions and the kinetic equations (Eqs. (2)–(4)), enabled predicting PV, $K_{232}$, and $K_{268}$ at specific time intervals (7, 14, 21, and 28 days). For light-protected oils [20], zero-, first-, and second-order kinetic models based on PV, $K_{232}$, and $K_{268}$ accurately described the progression of these quality parameters over the 28-day period, with root mean square error (RMSE) values ranging from 0.43 to 1.28 meq. $O_2$/kg, 0.087 to 0.263, and 0.004 to 0.007, respectively. Using Eqs. (5)–(7), SLs for light-protected oils under simulated household conditions were estimated at 33–49 days, 101–140 days, and 276–291 days based on $K_{232}$, PV, or $K_{268}$ data, respectively. Experimental findings suggested that the real SL of these oils exceeded 28 days, allowing to identify $K_{232}$ as the most conservative SL indicator, which aligns with the present study's findings for light-protected oils. In contrast, analysis of the data reported by Rodrigues et al. [18] for light-exposed oils, indicated that first- and second-order models for PV yielded satisfactory fits ($0.39 \leq RMSE \leq 1.97$ meq. $O_2$/kg), whereas

**Table 3** Mean (±standard deviation) intensities of gustatory sensations and of overall qualitative gustatory-retronasal sensations perceived in cv. Cobrançosa EVOO by a trained panel (0: not perceived to 10: maximum perceived intensity) over a 63-day time-period of simulated household consumption conditions (daily opening and shaken with oil weekly withdrawn) stored in darkness (light-protected oils) or exposed to natural and artificial light (light-exposed oils) at ambient temperature (18±2ºC)

| Gustatory sensation | Storage conditions | Storage time | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0 days | 7 days | 14 days | 21 days | 28 days | 35 days | 42 days | 49 days | 56 days | 63 days | P-value[#] |
| Positive sensations | | | | | | | | | | | | |
| Ripe fruit | Light | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | 5.3±0.3bA | 6.5±0.3aA | N.E | N.E | 0.0004 |
| | Dark | | N.D. | N.D. | N.D. | N.D. | N.D. | 3.3±0.2bB | 5.6±0.3aB | 5.6±0.7a | 5.7±0.8a | <0.0001 |
| | P-value[§] | – | – | – | – | – | – | <0.0001 | 0.0021 | – | – | – |
| Green fruity | Light | 7.0±0.5a | 6.4±0.2aA | 5.2±0.3bB | 5.1±0.2bA | 4.8±0.3bA | 3.4±1.2cA | N.D. | N.D. | N.E | N.E | <0.0001 |
| | Dark | | 6.4±0.2bA | 5.8±0.2cA | 4.7±0.3 dB | 2.5±0.3eB | 2.3±0.2eA | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.8390 | 0.0107 | 0.0284 | <0.0001 | 0.0859 | – | – | – | – | – |
| Sweet | Light | 1.5±0.2d | 1.6±0.2dA | 2.2±0.3dA | 4.1±0.6cA | 4.1±0.4cA | 4.4±0.7bcA | 5.4±0.8abA | 5.4±0.4aA | N.E | N.E | <0.0001 |
| | Dark | 1.5±0.2e | 1.7±0.2eA | 2.2±0.2eA | 3.3±0.5dA | 4.1±0.4cdA | 4.5±0.3bcA | 5.4±0.4abA | 5.8±0.5aA | 6.2±1.1ab | 6.3±0.1a | <0.0001 |
| | P-value[§] | – | 0.263 | 0.946 | 0.0619 | 0.9999 | 0.695 | 0.842 | 0.281 | – | – | – |
| Bitter | Light | 5.7±0.3a | 5.3±0.3aB | 5.1±0.6aA | 3.9±0.8bA | 3.6±0.4bA | 1.4±0.2cB | 1.3±0.5cA | N.D. | N.E | N.E | <0.0001 |
| | Dark | | 5.7±0.1aA | 5.2±0.1bA | 3.9±0.2cA | 3.7±0.1cA | 2.8±0.3dA | 1.2±0.4eA | 0.9±0.1ef | 0.6±0.2f | 0.7±0.1ef | <0.0001 |
| | P-value[§] | – | 0.0251 | 0.7040 | 0.9999 | 0.5360 | <0.0001 | 0.6230 | – | – | – | – |
| Pungent | Light | 6.3±0.3a | 6.1±0.2aA | 5.8±0.4aA | 5.5±0.3aA | 4.5±0.4bA | 2.2±0.5cA | 0.8±0.3 dB | N.D. | N.E | N.E | <0.0001 |
| | Dark | | 6.1±0.4aA | 5.9±0.2aA | 4.2±0.3bB | 3.6±0.2bB | 2.8±0.4cA | 2.1±0.7dA | 0.9±0.2eA | 0.7±0.1e | 0.7±0.1e | <0.0001 |
| | P-value[§] | – | 0.7210 | 0.7530 | <0.0001 | 0.0024 | 0.0859 | 0.0055 | – | – | – | – |
| Apple | Light | 5.3±0.7a | 4.6±0.1abA | 4.6±0.4abA | 4.3±0.3bcA | 3.8±0.2bcdA | 3.6±0.4cdA | 3.1±0.3deB | 2.5±0.4eB | N.E | N.E | <0.0001 |
| | Dark | | 4.8±0.4abA | 4.3±0.2bcA | 4.1±0.5bcA | 4.0±0.2bcA | 3.9±0.3cA | 3.8±0.3cA | 3.7±0.6cA | 3.6±0.4c | 3.5±0.4c | <0.0001 |
| | P-value[§] | – | 0.2080 | 0.2540 | 0.6750 | 0.1530 | 0.2060 | 0.0065 | 0.0038 | – | – | – |
| Tomato | Light | 6.5±0.4a | 5.8±0.2aA | 5.2±0.1cB | 4.1±0.2dA | 3.9±0.3dA | 3.3±0.2eA | N.D. | N.D. | N.E | N.E | <0.0001 |
| | Dark | | 6.0±0.3abA | 5.6±0.2bA | 3.9±0.5cA | 3.7±0.2cA | 2.8±0.1 dB | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.3150 | 0.0121 | 0.4330 | 0.2080 | 0.0047 | – | – | – | – | – |
| Dry fruits | Light | 3.1±0.2c | 3.8±0.2bcA | 3.8±0.3bcA | 4.0±0.5bA | 4.5±0.4bA | 5.5±0.6aA | 5.5±0.5aA | 5.6±0.4aA | N.E | N.E | <0.0001 |
| | Dark | 3.1±0.2d | 3.6±0.5cdA | 3.7±0.4cdA | 3.4±0.6dA | 3.8±0.4cdB | 4.3±0.2bcB | 4.7±0.3abB | 4.9±0.2abB | 5.4±0.5a | 5.4±0.2a | <0.0001 |
| | P-value[§] | – | 0.5230 | 0.6540 | 0.1640 | 0.0109 | 0.0055 | 0.0147 | 0.0099 | – | – | – |
| Tomato leaves | Light | 6.0±0.8a | 4.1±0.2bB | 3.4±0.3bcA | 3.4±0.1cA | 2.3±0.3 dB | N.D. | N.D. | N.D. | N.E | N.E | <0.0001 |
| | Dark | | 4.6±0.4bA | 3.7±0.3cA | 3.5±0.2cdA | 3.7±0.3cA | 2.8±0.2d | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.0481 | 0.1040 | 0.0885 | <0.0001 | – | – | – | – | – | – |
| Cabbage | Light | 6.2±0.7a | 5.1±0.2bA | 4.6±0.3bA | 3.7±0.4cA | 2.2±0.3dA | N.D. | N.D. | N.D. | N.E | N.E | <0.0001 |
| | Dark | | 5.5±0.4aA | 4.5±0.2bA | 3.5±0.3cA | 2.5±0.3dA | 1.7±0.2e | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.0896 | 0.3660 | 0.2680 | 0.1990 | – | – | – | – | – | – |
| Fresh grass | Light | 5.8±0.6a | 4.3±0.24bB | 4.0±0.4bB | 3.6±0.3bcA | 3.1±0.5cA | 2.3±0.1dA | N.D. | N.D. | N.E | N.E | <0.0001 |
| | Dark | | 5.0±0.21bA | 4.5±0.2bA | 3.8±0.1cA | 2.3±0.3 dB | 1.6±0.3eB | N.D. | N.D. | N.D. | N.D. | <0.0001 |
| | P-value[§] | – | 0.0007 | 0.0302 | 0.2740 | 0.0140 | 0.0008 | – | – | – | – | – |
| Dry grass | Light | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | 4.7±0.5aA | 5.1±0.6aA | N.E | N.E | 0.2780 |
| | Dark | | N.D. | N.D. | N.D. | N.D. | N.D. | 2.6±0.1cB | 4.5±0.4bA | 5.2±0.5a | 5.3±0.3a | <0.0001 |
| | P-value[§] | – | – | – | – | – | – | <0.0001 | 0.0838 | – | – | – |

**Table 3** (continued)

| Gustatory sensation | Storage conditions | Storage time | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0 days | 7 days | 14 days | 21 days | 28 days | 35 days | 42 days | 49 days | 56 days | 63 days | P-value[#] |
| Harmony | Light | 8.1±0.2ab | 8.0±0.2abA | 7.9±0.2bcA | 7.8±0.2bcA | 8.3±0.1aA | 7.9±0.2bcB | 7.9±0.2bcA | 7.6±0.2cA | N.E | N.E | <0.0001 |
| | Dark | | 7.7±0.3abA | 8.0±0.1aA | 7.8±0.2abA | 8.3±0.2aA | 8.1±0.1aA | 7.9±0.3abA | 7.2±0.7bA | 7.7±0.4ab | 7.6±0.4ab | 0.0002 |
| | P-value[§] | – | 0.0943 | 0.3770 | 0.9290 | 0.8530 | 0.0179 | 0.8590 | 0.2500 | – | – | – |
| Overall qualitative gustatory-retronasal sensations | | | | | | | | | | | | |
| Complexity | Light | 7.2±0.2a | 7.4±0.2aA | 7.2±0.2aA | 7.1±0.2aA | 6.6±0.4abB | 5.8±0.8bcB | 5.6±0.4cA | 3.8±0.8 dB | N.E | N.E | <0.0001 |
| | Dark | | 7.3±0.3aA | 7.2±0.3aA | 7.0±0.1abA | 7.3±0.5aA | 6.9±0.5abcA | 5.2±0.3eA | 5.6±0.9deA | 6.0±0.4cde | 6.2±0.5bcd | <0.0001 |
| | P-value[§] | – | 0.7510 | 0.9080 | 0.2800 | 0.0228 | 0.0040 | 0.1330 | 0.0089 | – | – | – |
| Persistence | Light | 8.2±0.5a | 8.3±0.2aA | 8.3±0.1aA | 8.1±0.3aA | 6.6±0.4bB | 6.8±0.4bA | 5.4±0.5cA | 3.7±0.6 dB | N.E | N.E | <0.0001 |
| | Dark | | 8.3±0.3aA | 8.2±0.2abA | 8.2±0.3abA | 7.4±0.4bcA | 6.9±0.5cdA | 5.2±0.4fA | 5.7±0.8efA | 6.0±0.5def | 6.2±0.4de | <0.0001 |
| | P-value[§] | – | 0.9999 | 0.7040 | 0.8020 | 0.0158 | 0.6630 | 0.3050 | 0.0021 | – | – | – |
| Negative sensations | | | | | | | | | | | | |
| Rancid | Light | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.E | N.E | – |
| | Dark | | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. | – |
| | P-value[§] | – | – | – | – | – | – | – | – | – | – | – |

N.D.: not detected; N.E.: not evaluated since an intense olfactory defect was perceived (intensity > 3.50, as reported in Table 2)

[#] One-way ANOVA: different lowercase letters at each household lighting storage condition and for each parameter indicate a statistically significant effect (P-value < 0.05) of the storage time-period (from 0 to 63 days), according to the multicomparison Tukey's test

[§] t-Student's test: different uppercase letters at each household storage time-period and for each parameter indicate a statistically significant effect (P-value < 0.05) of the two studied lighting conditions (dark versus light exposure)

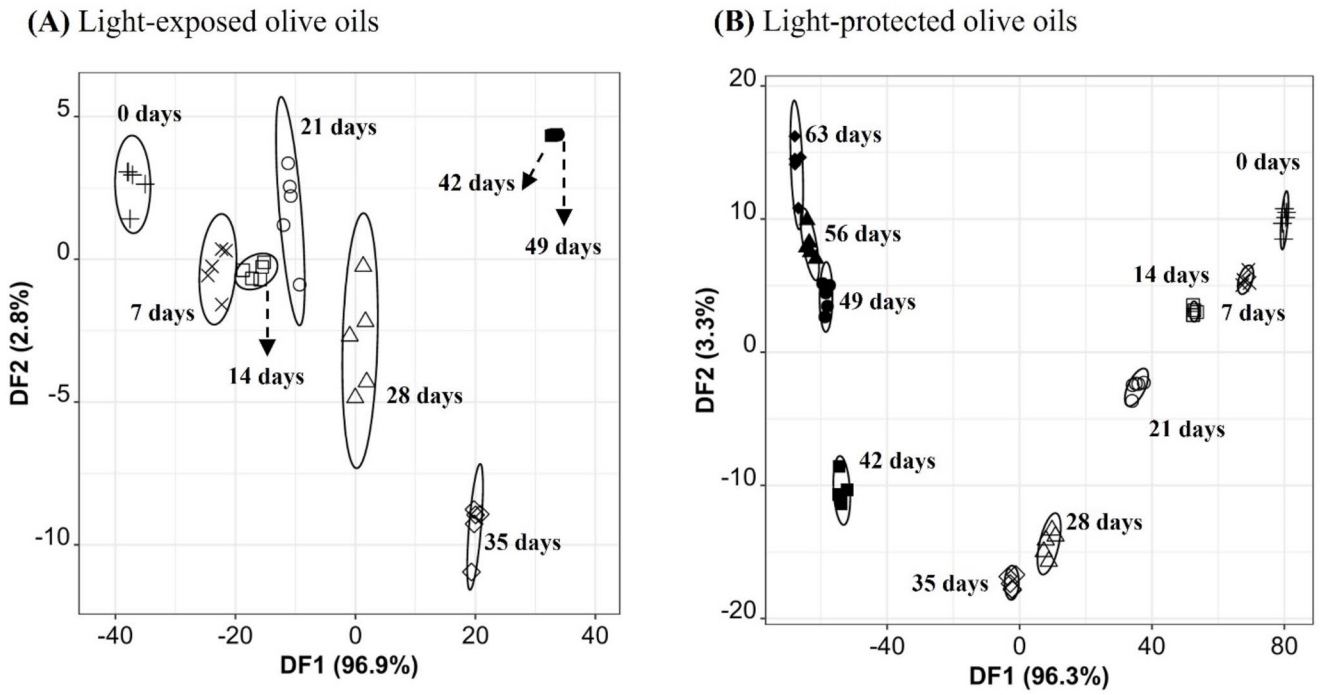



**Fig. 1** 2D-LDA plots of the two first discrimination functions (DF1 and DF2) regarding the supervised discrimination of cv. Cobrançosa olive oils according to the time-period of the simulated household consumption/storage conditions: (**A**) light-exposed oils; and (**B**) light-protected oils

the zero-order model significantly overestimated PV progression ($6.49 \leq RMSE \leq 6.72$ meq. $O_2$/kg). For $K_{232}$ and $K_{268}$ parameters, all kinetic models developed in this study (Table 4) could provide satisfactory fittings of the observed time-evolutions, with RMSE values ranging from 0.056 to 0.189 and from 0.014 to 0.028, respectively. Predicted SL for light-exposed oils, based on $K_{232}$, ranged from 19 to 48 days, consistent with the experimental SL of 21 to 28 days established using $K_{232}$ mean values reported by Rodrigues et al. [18]. PV- and $K_{268}$-based models, however, tended to overestimate SL, predicting values of 56–116 days and 101–173 days, respectively. Overall, this analysis confirms the applicability of the proposed kinetic modelling approach for SL prediction of olive oils under domestic storage conditions. The kinetic models, particularly the first- and second-order models with the respective *k* values derived in this study, accurately predicted the SL of light-protected oils (33–49 days) and light-exposed oils (19–48 days), closely aligning with experimental results from Klisović et al. [20] (>28 days) and Rodrigues et al. [18] (21–28 days), respectively. It is important to emphasize that while the kinetic models presented in this study were developed based on the time-evolution of quality data from cv. Cobrançosa EVOO, their successful application to EVOO extracted from other olive cultivars, specifically cvs. Arbequina, Istarska Bjelica, and Buža, clearly demonstrates their wider applicability.

## Conclusions

The study findings highlight the significant influence of household storage conditions on the overall quality of olive oils and the fast degradation occurring once opened. Oxidation is the main contributor to quality loss after bottle opening, particularly photo-oxidation, with preservation from light being the more relevant procedure to delay EVOO degradation, which could be doubled in comparison with bottles exposed to light. As to the time when EVOO declassification occurs, it is highly dependent on the initial characteristics of the EVOO itself. Therefore, fresher EVOO are expected to sustain the classification longer during use. Additionally, this study corroborated that the EVOO preserve their health claim associated with biophenols under the simulated conditions, even if exposed to light.

The results underscore the usefulness of time-to-reach-upper-limit (TRUL) models for predicting EVOO shelf life in glass bottles under typical household conditions and at constant ambient temperature, provided initial physico-chemical quality parameters are known, with PV providing the most accurate SL predictions for light-exposed oils and, $K_{232}$ together with a zero-order TRUL model, for light-protected oils consistent with the observed SL ranges. The kinetic-based modelling approach was further validated using literature data, allowing a satisfactory agreement between the predicted SL values and the experimental SL data.

**Table 4** Kinetic reaction rates ($k \pm$ standard error) estimated by linear regression and respective correlation coefficients (R) regarding zero-, first- or second-order kinetic models for PV, $K_{232}$ and $K_{268}$ based on the experimental data of cv. Cobrançosa EVOO collected during the 63-day time-period at 18 ± 2 ºC

| Kinetic models | Light-exposed oils | | |
|---|---|---|---|
| Zero-order | PV | $K_{232}$ | $K_{268}$ |
| R | 0.942 | 0.996 | 0.997 |
| $k$ | 0.48 ± 0.06 meq. $O_2$ $kg^{-1}$ $day^{-1}$ | $(99 \pm 3) \times 10^{-4}$ $day^{-1}$ | $(102 \pm 3) \times 10^{-5}$ $day^{-1}$ |
| SL | 20 ± 3 days | 70 ± 2 days | 60 ± 2 days |
| First-order | PV | $K_{232}$ | $K_{268}$ |
| R | 0.990 | 0.995 | 0.996 |
| $k$ | $(23 \pm 1) \times 10^{-3}$ $day^{-1}$ | $(48 \pm 2) \times 10^{-4}$ $day^{-1}$ | $(56 \pm 2) \times 10^{-4}$ $day^{-1}$ |
| SL | 27 ± 1 days | 73 ± 3 days | 58 ± 2 days |
| Second-order | PV | $K_{232}$ | $K_{68}$ |
| R | 0.991 | 0.993 | 0.994 |
| $k$ | $(126 \pm 8) \times 10^{-5}$ kg (meq. $O_2$)$^{-1}$ $day^{-1}$ | $(24 \pm 1) \times 10^{-4}$ $day^{-1}$ | $(31 \pm 1) \times 10^{-3}$ $day^{-1}$ |
| SL | 35 ± 2 days | 63 ± 3 days | 56 ± 2 days |
| Kinetic models | Light-protected oils | | |
| Zero-order | PV | $K_{232}$ | $K_{268}$ |
| R | 0.985 | 0.977 | 0.966 |
| $k$ | $(132 \pm 8) \times 10^{-3}$ meq. $O_2$ $kg^{-1}$ $day^{-1}$ | $(14 \pm 1) \times 10^{-3}$ $day^{-1}$ | $(36 \pm 3) \times 10^{-5}$ $day^{-1}$ |
| SL | 71 ± 4 days | 49 ± 4 days | 171 ± 14 days |
| First-order | PV | $K_{232}$ | $K_{268}$ |
| R | 0.991 | 0.971 | 0.966 |
| $k$ | $(96 \pm 5) \times 10^{-4}$ $day^{-1}$ | $(68 \pm 6) \times 10^{-4}$ $day^{-1}$ | $(22 \pm 2) \times 10^{-4}$ $day^{-1}$ |
| SL | 66 ± 3 days | 48 ± 4 days | 149 ± 14 days |
| Second-order | PV | $K_{232}$ | $K_{268}$ |
| R | 0.993 | 0.965 | 0.966 |
| $k$ | $(71 \pm 3) \times 10^{-5}$ kg (meq. $O_2$)$^{-1}$ $day^{-1}$ | $(32 \pm 3) \times 10^{-4}$ $day^{-1}$ | $(13 \pm 1) \times 10^{-3}$ $day^{-1}$ |
| SL | 62 ± 3 days | 48 ± 5 days | 135 ± 10 days |

Shelf-life (SL) in terms of EVOO classification was predicted based on TRUL models

Further research involving a broader range of EVOOs of varying cultivars, geographical origins, and particularly with initial chemical compositions and under different household consumption and storage conditions will be needed to validate this predictive approach further and estimate reaction kinetic parameters as reliable as possible. Indeed, to address the limitation of this study regarding simulated home temperatures, it would be necessary to extend the research to a broader range of typical household temperatures. This would better represent olive oil usage across different seasons, from winter to summer, and enhance the applicability of the developed kinetic models by incorporating an Arrhenius-type temperature dependence for the quality degradation reaction rates. Similarly, it would be useful to extend the present study by exploring, besides the typical glass bottles, new packaging solutions including the use of intelligent/smart packages and their impacts on minimizing the olive oil quality degradation during its household consumption.

**Author contributions** S.A.: data curation, formal analysis, investigation, writing - original draft, writing - review & editing; N.R: supervision, resources, funding acquisition, methodology, writing - review & editing; A.C.A.V.: conceptualization, resources, formal analysis, writing - review & editing; R.C: data curation, writing - review & editing; M.K.: supervision, writing - review & editing; J.A.P.: conceptualization, methodology, writing - review & editing; S.C.: data curation, supervision, resources, funding acquisition, writing - review & editing; A.M.P.: supervision, methodology, formal analysis, validation, writing - original draft, writing - review & editing.

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**Data availability** Data is provided within the manuscript or supplementary information files.

## Declarations

**Conflict of interest** The authors declare no conflict of interest.

**Consent to participate** All panellists that are members of the Olive oil Sensory Panel of Escola Superior Agrária of Instituto Politécnico de Bragança (Portugal), gave their written informed consent to participate in this study.

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