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Assessing the Shelf-Life of Olive Oil Under Different Storage Conditions: A Review of Predictive Models

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Abstract
Olive oil holds a significant position in the global vegetable oil market, often reaching high prices compared to other vegetable oils. However, like other oils, it is vulnerable to oxidation, which can degrade its quality during storage, making it essential to determine its shelf-life. So, kinetic or empirical models have been developed to estimate how long olive oil can maintain the legal quality standards necessary for its commercial classification or to be marketed with nutritional or health claim. This study reviews recent advancements in modelling approaches to predict the shelf-life of olive oil under different storage conditions, namely storage duration (from 2 months to 2 years), temperature (20–50 ºC), and light exposure (light versus dark storage). Most models estimate the timeframe in which olive oil remains compliant with regulatory requirements for specific commercial grades, namely extra virgin olive oil, with fewer models addressing health-related claims. Developed models include pseudo zero-, pseudo first-, and pseudo second-order kinetic models and empirical models, derived from experimental data on the oil's chemical stability over time. While empirical models can be highly accurate, they often require extensive chemical data, including for compounds for which no legal thresholds exist, and complex statistical techniques, limiting their use by non-specialists. In contrast, kinetic models offer simpler and user-friendly mathematical equations. Nonetheless, olive oil's shelf-life predictions remain influenced by factors such as initial oil composition, packaging materials, and storage conditions, underscoring the ongoing need to refine the predictive models.

Keywords Kinetic-based models · Empirical-based models · Storage conditions · Commercial category · Nutritional claims · Health claims

Introduction

The Mediterranean diet is widely recognized for emphasizing olive oil as one of the main elements [1]. Extra virgin olive oil (EVOO) is highly appreciated by consumers for its richness in monounsaturated fatty acids and natural antioxidants, offering numerous health benefits [2, 3]. The main reported benefits from EVOO consumption are linked to their antioxidant, anti-inflammatory, and anti-cancer properties, as well as the potential prevention of cardiovascular and neurodegenerative diseases [4, 5]. The high concentration of fatty acids in EVOO triggers enzymatic reactions [6, 7] that lead to auto-oxidation or, if the oils are exposed to light, to photo-oxidation. It should be remarked that primary oxidation products are odourless and flavourless, precursors of unpleasant odours and flavours, that may arise in oils due to auto- or photo-oxidation reactions. These oxidation processes generate unwanted volatile compounds (secondary oxidation products) leading to olfactory and/or gustatory-retronasal defects [7–9]. As a result, the quality and economic value of the oil diminishes, preventing its further commercialization as EVOO. Other factors influencing olive oil quality include the healthy level of the fruit and its ripening level, as well as post-harvest practices like storage conditions before processing [10], mechanical extraction techniques, thermo-batching conditions [11], packaging methods [12], and storage conditions after extraction [13]. The European

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Regulation encompasses various commercial categories for olive oil and defines the legal limits of quality parameters for each category [14]. In which concerns the highest value category, oil can only be labelled as EVOO if, simultaneously, the free acidity (FA) is not greater than 0.8% of oleic acid; the peroxide value (PV) is lower or equal to 20 mEq O_2 kg^{-1}; the extinction coefficients at 232 nm (K_{232}) and 268 nm (K_{268}) are lower or equal to 2.5 and 0.22, respectively. Besides, from a sensory point of view, it is required that fruity green or ripe sensation is perceived and that no sensory olfactory or gustatory defect is detected. The time evolution of these chemical-sensory parameters during storage is related to oxidation reactions, allowing assessing the oils' oxidation level. Additionally, other compositional parameters, including, for example, waxes and ethylic ethers, must also fulfil legal thresholds, but their levels and time-evolution are not strictly related to oxidation. Overall, maintaining and ensuring the stability of EVOO is vital for both producers and consumers [15].

The term "shelf-life" (SL) for EVOO refers to the time duration in which it maintains its flavour (positive olfactory and gustatory sensations and the absence of sensory defects) and quality parameters (such as acidity, peroxide value, specific extinction coefficients, sensory attributes, fatty acid ethyl esters and waxes) within legal limits under typical storage conditions [16, 17]. Additionally, the SL concept may be extended to include the time durability of a specific nutritional or health claim (e.g., polyphenolic content, vitamin E content or unsaturated fatty acids relative abundance), in case these claims are included in the EVOO label. As highlighted by Roselli et al. [18], labelling that specifies health claims based on the polyphenol content in olive oil could serve as an effective indicator of both the "highest quality" and "healthiest" Italian EVOO. Providing consumers with accurate information about the expected SL is crucial, considering factors such as packaging materials, temperature fluctuations, and exposure to light and air during transportation or storage at the different actors of the commercial chain (e.g., producer, retailer, and supermarket). Shelf-life testing can be conducted in real-time or through accelerated shelf-life testing (ASLT). ASLT accelerates the oxidation process, allowing predicting the SL of EVOO through extrapolation [19]. However, the accuracy of SL predictions using ASLT may be compromised, as the values obtained may overestimate the actual shelf-life of EVOO [20]. The limitations of ASLT methodologies for estimating the SL of food products have been well-documented and recognized long ago [21]. Therefore, conducting real-time studies is crucial for more precise estimations [12]. Analytical data obtained from either real-time or ASLT conditions can be used, independently or merged, to develop models for predicting the SL of EVOO [22].

Olive Oil Oxidation and Antioxidant Activity with a Mechanism Emphasis

The complexity of oxidation processes that take place in food matrices, namely in olive oils, is often disregarded and excessively simplified by not integrating multiple oxidation markers, stages and/or approaches. Moreover, to better understand olive oil oxidation, it is essential to consider both oxidation processes, especially of unsaturated fatty acids, and the antioxidant effectiveness of several minor components like phenols, phytosterols, carotenoids, tocopherols and vitamins. Also, the role played by chlorophylls, free fatty acids and transition metals, among other oil's constituents, in the oxidation/antioxidation/pro-oxidation reactions is essential. Two types of oxidation processes occur, namely auto-oxidation (in dark conditions) and photo-oxidation (under light exposure). The extension of the former is related to the type and content of unsaturated fatty acids, requiring the latter the presence of photosensitizers and singlet oxygen quenchers. In this latter case, the literature refers that polar phenols that act as chain-breaking antioxidants, play a rather limited role during exposure to light [23]. Besides, enzymatic oxidation may also take place due to the presence of some enzymes in the olive oil [24]. Regarding auto-oxidation, phenols like o-diphenol hydroxytyrosol and its oleosidic forms contribute to olive oil stability due to their antioxidant properties. Similarly, tocopherols, namely α-tocopherol, enhance the oil's stability by donating a hydrogen atom to chain-propagating peroxyl radicals, thus limiting the auto-oxidation reactions. Its capability to quench singlet oxygen by a charge transfer mechanism also contributes to decreasing the level of oils' photo-oxidation. However, the fast degradation of α-tocopherol during light exposure of olive oil restrings its antioxidant role in the initial stages of oxidation [23, 25]. On the other hand, chlorophyll pigments and derivatives have pro-oxidative effect, acting as photosensitizers due to the ability to transfer energy from light to triplet oxygen, producing thus singlet oxygen, which then reacts with the unsaturated fatty acids. Oppositely, carotenoids inhibit photo-oxidation by quenching singlet oxygen and triplet excited states of photosensitizers. The physical quenching mechanism of carotenoids is based on their low singlet energy state, which facilitates the acceptance of energy from singlet oxygen. Additionally, the antioxidant activity of carotenoids is related to a light-filtering effect due to the extended conjugation system [23].

The oxidation mechanisms of edible oils, such as olive oil, are well-documented [12, 26]. This section outlines those mechanisms.

Auto-oxidation involves free radical chain reactions with initiation, propagation, and termination steps [27], as illustrated in Fig. 1.

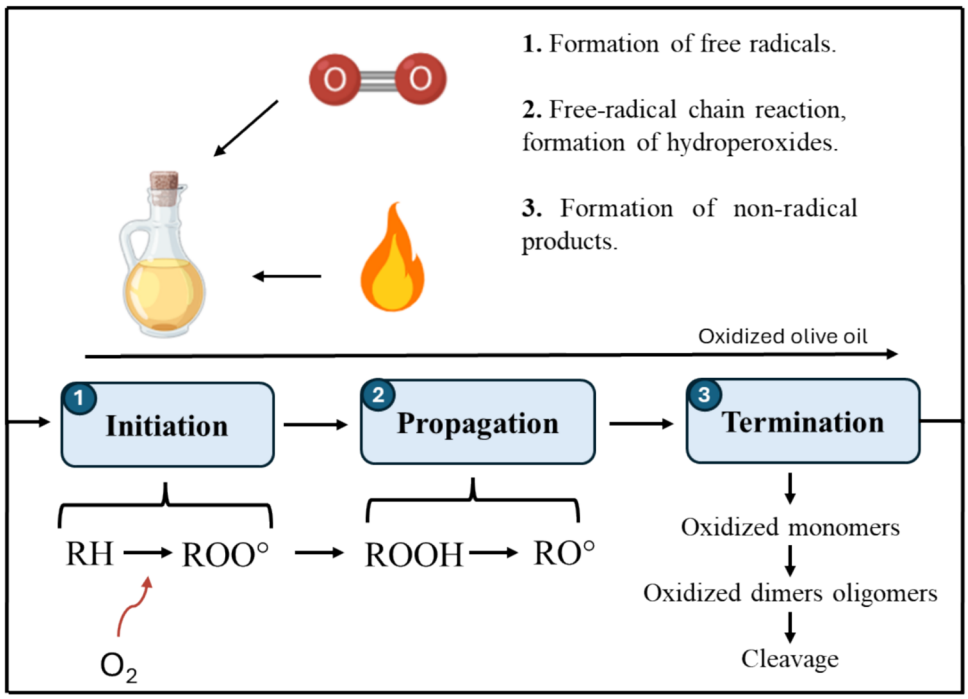

Fig. 1 Auto-oxidation schematic representation

During initiation, hydrogen atoms are removed from the methylene carbon of fatty acids or acylglycerols, forming free lipid alkyl radicals, which are accelerated by light, heat, metal catalysis, and UV/Vis light. In the propagation step, lipid alkyl radicals react with atmospheric triplet oxygen to produce lipid peroxy radicals. These radicals then abstract hydrogen from other lipid molecules, forming lipid hydroperoxides and new alkyl radicals, which are the primary oxidation products. During termination, lipid peroxy radicals react with alkyl radicals or with each other. At high temperatures or in the presence of metals, lipid hydroperoxides decompose into alkoxy radicals, forming aldehydes, ketones, acids, esters, alcohols, and short-chain hydrocarbons through homolytic cleavage, which may undergo further oxidation, leading to off-flavour compounds [28].

Light exposure accelerates oxidation in the presence of photosensitizers such as chlorophylls through photosensitized oxidation, which follows the singlet oxygen pathway [27], as schematically shown in Fig. 2, in contrast to the radical-driven mechanism of auto-oxidation.

Photosensitizers absorb light and change from a singlet to an excited triplet state (intersystem crossing), producing radicals or reacting with atmospheric triplet oxygen to form superoxide anions via electron transfer. These anions suffer spontaneous dismutation leading to the formation of hydrogen peroxide, which reacts with superoxide to produce singlet oxygen in the presence of metals like iron or copper. The excitation energy of triplet sensitizers is transferred to atmospheric triplet oxygen, forming singlet oxygen via a triplet–triplet annihilation mechanism. This singlet oxygen

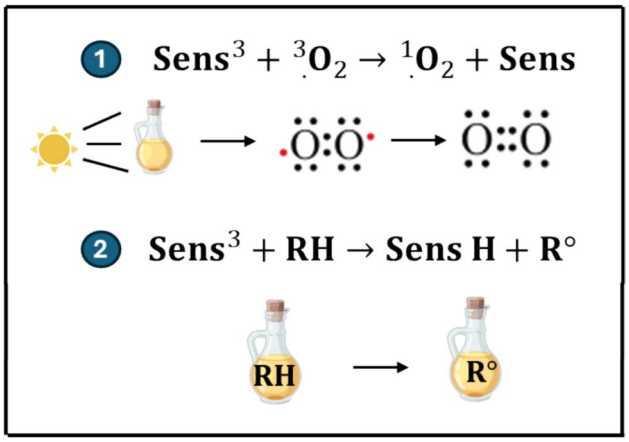

Fig. 2 Photo-oxidation schematic representation

reacts with unsaturated fatty acids, forming alkyl hydroperoxides through an ene reaction. The hydroperoxides formed cause double bond migration and the formation of trans fatty acids, resulting in a mixture of conjugated and non-conjugated hydroperoxides. These hydroperoxides decompose similarly to those formed in auto-oxidation, producing undesirable oxidation products.

Additionally, oxidation can also occur when triggered by enzymes, particularly lipoxygenase [27], especially when the olive fruit is physically damaged during harvesting, transportation or mechanical processing (Fig. 3). Triglycerides are hydrolysed by lipase through the lipoxygenase pathway, producing monohydroperoxides. Specifically, lipoxygenase

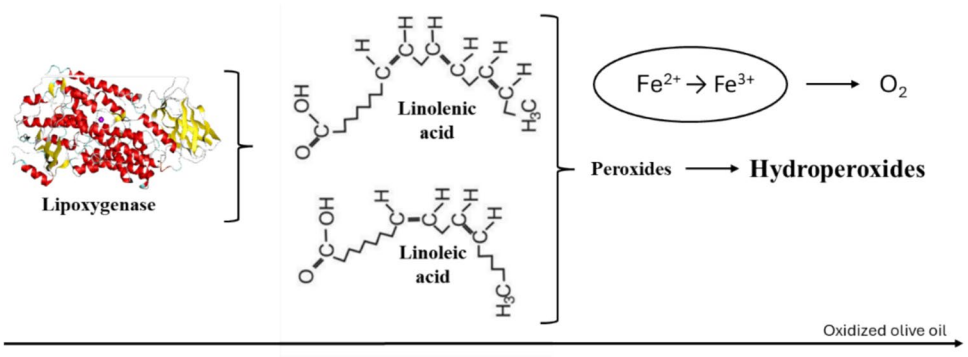

Fig. 3 Enzymatic-oxidation schematic representation

acts on linolenic and linoleic acids, producing peroxides and hydroperoxides with conjugated double bonds [28]. This oxidation process involves three steps. First, lipoxygenase allows the transfer of hydrogen atoms from linoleic acid to the coenzyme Fe(III)-OH, generating radicals and reducing the Fe^{3+} of lipoxygenase to an inactive Fe^{2+} state. Then, the radicals combine with oxygen, creating peroxide radicals and producing O_2 during the process. Finally, the peroxide radicals are reduced by the Fe^{2+} of lipoxygenase forming hydroperoxides, and the enzyme is reactivated as Fe^{3+} [29]. The primary oxidation products are precursors to unpleasant aromas and tastes. In summary, monohydroperoxides are first converted into aldehydes by lyase, then into alcohols by alcohol dehydrogenase, and subsequently transformed into acetate compounds by alcohol acetyl transferase [6, 7]. Additionally, polyphenolic compounds break down due to the action of polyphenol oxidase and peroxidase [6], producing undesirable volatile compounds such as alkanes, alkenes, aldehydes, and ketones. These secondary oxidation products contribute to sensory defects like rancidity [7–9].

Tocopherols and carotenoids have been extensively studied for their antioxidant effects and reaction mechanisms [26]. Tocopherols compete with unsaturated fats for lipid peroxy radicals, scavenging them by donating a hydrogen atom, which forms lipid hydroperoxides and stable tocopheroxy radicals. This process can slow oil oxidation during the propagation phase of auto-oxidation. However, at high concentrations, tocopherols may act as pro-oxidants, a process known as tocopherol-mediated peroxidation. Additionally, tocopherols can reduce oil oxidation under light by quenching singlet oxygen, either physically or chemically. Carotenoids slow oil oxidation through light filtering, singlet oxygen quenching, sensitizer inactivation, and free radical scavenging. Quenching occurs mainly via energy transfer from singlet oxygen to carotenoids, dissipating the energy as heat. Carotenoids can absorb energy from excited sensitizers. While they rarely donate hydrogen to alkyl or peroxy radicals of unsaturated fatty acids, they can donate hydrogen to hydroxyl radicals, forming stable carotene radicals that react with other radicals to form non-radical products at low oxygen levels. At high oxygen levels, carotene peroxy radicals can propagate lipid oxidation. Carotenoids can also donate electrons to free radicals, forming cation radicals that may react with alkyl, alkoxy, or peroxy radicals during oil oxidation. However, carotenoids can also act as pro-oxidants by degrading hydroperoxides into hydroxyl- or epoxycarotenes.

Predicting the Shelf-Life of Olive Oils Using Different Empirical and/or Kinetic Based Strategies

Two types of prediction models are commonly employed: empirical or kinetic models. Empirical models estimate the shelf-life by considering stability parameters [30]. The empirical models often rely on the use of multivariate statistical tools, namely multiple linear regression models (MLRM) for explaining the relationship between shelf-life (SL), initial composition and/or oxidation progress, being the independent variables included in the final MLRM usually identified by applying a heuristic or meta-heuristic variable selection algorithm:

$$SL = a_0 + a_1 \times X_1 + a_2 \times X_2 + \cdots + a_n \times X_n \quad (1)$$

where:

- SL is dependent variable,
- X_i are the independent variables, and
- a_i are the regression coefficients obtained by regression analysis, having a negative sign if the related independent variable has a negative impact on the oil's SL, contributing to its decreasing, and a positive sign if the related independent variable positively contributes to increase the oil's SL.

In contrast, kinetic models rely on estimating reaction rates (k) for key physicochemical parameters, assuming a reaction pseudo order ($n \geq 0$) influenced by factors like storage time (t), temperature, and light. Equation (2) establishes a relationship between the content/level of a specific parameter of interest (C) and time (t). The positive or negative sign ($\pm$) in the equation reflects the increasing or decreasing trend

of the parameter over time, respectively. This equation is a crucial component in kinetic-based models.

$$\frac{dC}{dt} = \pm k \times C^n \tag{2}$$

The integration of Eq. (2), at a constant storage temperature, from the initial storage conditions ($t=0$ and $C=C_0$) and the current conditions (t and C) will result in various models based on the assumed reaction order. It should be noted that,

Pseudo zero-order reaction ($n=0$):

$$\frac{dC}{dt} = \pm k \rightarrow \int_{C_0}^{C} dC = \pm k \int_{0}^{t} dt \rightarrow C - C_0 = \pm k \times t \tag{3}$$

Pseudo first-order reaction ($n=1$):

$$\frac{dC}{dt} = \pm k \times C \rightarrow \int_{C_0}^{C} \frac{dC}{C} = \pm k \int_{0}^{t} dt \rightarrow ln\left(\frac{C}{C_0}\right) = \pm k \times t \tag{4}$$

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

$$\frac{dC}{dt} = \pm k \times C^n \rightarrow \int_{C_0}^{C} \frac{dC}{C^n} = \pm k \int_{0}^{t} dt \rightarrow \frac{C^{-n+1}}{-n+1} - \frac{C_0^{-n+1}}{-n+1} = \pm k \times t \tag{5}$$

By employing experimental data (t, c) and using Eqs. (3) to (5), one can ascertain the kinetic reaction rates at a constant temperature through linear regression analysis, being k equal to the linear slope. On the other hand, if wanted, the temperature dependence of the k with temperature, k(T), is usually described by an Arrhenius-type equation [31]:

$$k(T) = k_0 e^{\left(-\frac{E_a}{RT}\right)} \tag{6}$$

being k_0 a constant (sometimes designated as the frequency factor), E_a the activation energy, R the molar gas constant and T the absolute temperature.

However, this relationship assumes the existence of a linearity between the logarithmic of k, $ln(k)$, and $\frac{1}{T}$, which depending on the size of the database, may lead to positive or negative deviations to the Arrhenius mechanism (sub- or super-Arrhenius behaviours, respectively [32–35]. In this case, a modified equation can be used [36]:

$$k = k_0 T^n e^{-\frac{E_a}{RT}} \tag{7}$$

where k_0, n ($0<n<1$) and E_a are determined using nonlinear fitting techniques.

Depending on the specific parameter being investigated, two distinct types of kinetic models can subsequently be employed to assess or predict the shelf-life of olive oils (Mancebo-Campos et al. [17, 36]:

- time to reach the legal upper limit (TRUL), which is applied when the content/level of the parameter under study increases with the storage time (e.g., FA, PV, K_{232} or K_{268}); and,
- time to reach the legal lower limit (TRLL), which is applied when the content/level of the parameter under study decreases with the storage time (e.g., polyphenols content, or vitamin E content related to health or nutritional claims of olive oils, respectively), also referred in the literature as the time to lose the health claim (TLHC).

The SL can be determined by solving Eqs. (3) to (5) with respect to time 't,' resulting in the shelf-life. It is important to note that 'C' represents the maximum or minimum legal content/level of the parameter being evaluated for TRUL or TRLL models, respectively:

Pseudo zero-order reaction ($n=0$):

$$SL = \frac{C_{legallimit} - C_0}{\pm k} \tag{8}$$

Pseudo first-order reaction ($n=1$):

$$SL = \frac{ln\left(\frac{C_{legallimit}}{C_0}\right)}{\pm k} \tag{9}$$

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

$$SL = \frac{\frac{C_{legallimit}^{-n+1}}{-n+1} - \frac{C_0^{-n+1}}{-n+1}}{\pm k} \tag{10}$$

Li and Wang [22] examined models for the shelf-life of olive oil in a comprehensives review, overviewing the literature data published before 2018. However, given the academic and industrial significance of this subject, a new review has been conducted to compile the most recent developments in the field. The focus is on studies published from 2018 onwards, emphasizing their novelty, limitations, and advantages. Additionally, a critical assessment is undertaken to bring attention to discrepancies in reported values for the shelf-life of olive oil. The review also discusses potential reasons for these variations and explores the possibility of pointing out the most reliable physicochemical parameter for predicting the oil's shelf-life.

Prediction of EVOO Shelf-Life: Literature Reported Modelling Approaches

To predict EVOO shelf-life, the works published between 2018 and 2023, describe strategies that simulate various storage conditions, including light exposure versus dark storage and temperature (ranging, in general, from 15 to 60 ºC) storage conditions. Additionally, different packaging materials were considered, including transparent/amber glass or polyethylene terephthalate (PET) plastic bottles. The studies also considered open or closed bottles with volumes ranging from 40 to 250 mL. These factors are known to influence the level and progression of oxidation of edible oils, namely olive oil. For example, energy inputs from light trigger photo-oxidation, and heat is responsible for increasing thermal oxidation at higher storage temperatures. Also, the type of oxygen present (atmospheric triplet oxygen and singlet oxygen) and their availability due to the packaging headspace or gas permeability conditioned the different chemical mechanisms, auto-oxidation and photosensitized oxidation, which are responsible for the oxidation of olive oils. If oxygen is available, primary oxidation reactions are favoured, resulting in an increase in the peroxide value. Conversely, when oxygen access is limited, secondary oxidation reactions become predominant. This leads to the consumption of primary reaction products and increases the levels of extinction coefficients, particularly K_{268}. Apart from the energy input (e.g., light or heat) and the type of oxygen, the initial composition of the oil at the time of packaging, especially the fatty acid profile and its unsaturated fraction, as well as minor compounds such as metals (e.g., iron and copper), chlorophylls/pigments, phospholipids, free fatty acids, mono- and diacylglycerols, thermally oxidized compounds, and antioxidants (e.g., phenolic compounds, tocopherols, and carotenoids), also significantly influence the oxidation process of olive oil. The minor compounds can act as either antioxidants or pro-oxidants during storage [26].

In this sense, both kinetic and empirical modelling strategies were described for predicting the SL of EVOO, with a preference for the former. In the literature it is also possible to find at least one study merging both approaches, proposing a fused kinetic-empirical model. The following sections provide a brief overview of each study, aiming to update the reader about the recent advancements in this specific field. Despite the experimental and modelling efforts, it is important to acknowledge that the literature has so far only introduced a restricted number of mathematical models. Most of these models are built upon the conventional kinetic approach, as outlined in Eq. (2), or the empirical approach, based on Eq. (1).

Determining the SL of EVOO involves several key steps. Figure 4 provides a concise overview of the different stages that are essential for assessing the SL of EVOO.

The initial step entails identifying the chemical, physical, or biological parameter that should be monitored due to its association with the deterioration of the oil's quality or commercial grade. Subsequently, acceptable limits must be defined, often aligning with legal thresholds established for specific quality grade parameters (e.g., FA, PV, K_{232}, or K_{268}) or for compounds/families associated with recognized nutritional (e.g., minimum relative abundance of unsaturated fatty acids or vitamin E per stipulated amount of olive oil) or health claims (e.g., minimum polyphenol content per stipulated amount of olive oil). Following this, the storage conditions to be evaluated are chosen, whether under real-time conditions or ASLT conditions. In cases of real storage conditions, changes in oil quality are monitored under typical storage conditions, simulating those encountered in the commercialization chain of olive oil. When ASLT conditions are employed, deterioration reactions are accelerated, resulting in a shorter storage time compared to real conditions. Accelerated stability modelling comprises three key steps. Initially, it estimates the degradation rate at high temperatures (above the usual ambient storage conditions, in general, at more than 100 ºC). Following that, the next step involves establishing the relationship between the degradation rate and the temperature, which, in general, is of a logarithmic type. Lastly, the model predicts the SL of the oil at the desired storage temperature, usually ambient temperature (lower than 30 ºC), by extrapolation. However, the predictions are subject to the usual uncertainties associated with any extrapolation procedure. The final stage involves the development and application of models to estimate or predict the SL of EVOO, as outlined by Calligaris et al. [31].

Table 1 provides a summary of the most recent papers (from 2018 and 2023) discussed in this review, including key details regarding olive oil samples and storage conditions, chemical/sensory analysis, and statistical approach and modelling strategy applied to estimate the SL.

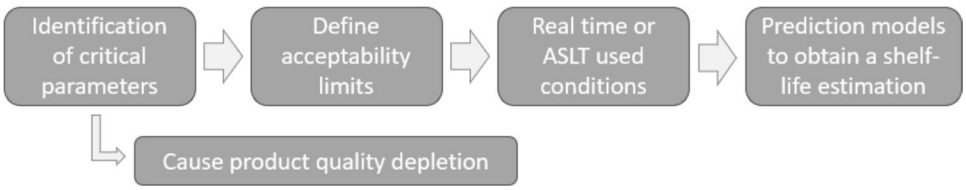

Fig. 4 Steps to do the determination of the olive oil's shelf-life

Table 1 Summary of SL prediction models discussed in the review covering the time range from 2018 and 2023

Olive oil sample	Storage conditions			Experimental assays		Statistical analysis	SL models	Reference
	Packaging material	Temperature	Time-period	Physical and Chemical parameters	Sensory analysis			
Kinetic modelling strategies								
Italian EVOO	(i) Transparent glass bottles (ii) Dark conditions	25, 40, 50 and 60 ºC	300 days	FA, PV, K_{232}, K_{270} Fatty acids profile Total phenols Tocopherols Pyropheophytin a Volatile compounds	n.p	(i) Bartlett's test (ii) One way ANOVA with Tukey´s HSD test (iii) Linear Regression	Pseudo zero-order kinetic models Arrhenius equation TRUL models	Conte et al. [37]
Spanish EVOO of cv. Cornicabra (industrial and Abencor extraction)	(i) Open amber glass bottles (ii) Dark conditions	25, 40, 50 and 60 ºC	19–93 weeks	PV, K_{232}, K_{270} Fatty acids profile Phenolic compounds Tocopherols Polyphenolics health claim content	n.p	(i) Linear regression models (ii) PCA (iv) One-way ANOVA with Tukey test	Pseudo zero-order and pseudo first-order models Arrhenius equation TRUL models TLHC (or TRLL) models	Mancebo-Campos et al. [17]
Italian EVOO (blend samples)	(i) Dark glass bottles (250 mL) (ii) Dark conditions	25, 40, 50 and 60 ºC	300 days	PV, K_{232}, K_{270} Phenolic compounds Tocopherols Pyropheophytin	Yes	n.p	ASLT method Pseudo zero-order models TRUL models	Calligaris et al. [19]
EVOO of two different cultivars	(i) Dark glass bottles	60 ºC	8 weeks	FA, PV, K_{232}, K_{270} Saponification value Fatty acid profile Tocopherols Sterols Oxidative stability	n.p	n.p	Thermodynamic modelling Reaction constants calculated from OS (or induction time) values determined using the Rancimat method SL determined based on OS temperature dependence equations	Gagour et al. [48]
Empirical modelling strategies								
EVOO from cvs. Biancolilla, Carolea, Coratina, Dolce di rossano, Frantoio, Nocellara del Belice and Nocellara Etnea, with different initial contents of natural antioxidants	(i) Green glass bottles	15–18ºC	12 months	FA, PV, K_{232}, K_{270} Fatty acid profile Phenolic compounds Tocopherols Volatile compounds Sterols Ethyl esters 1,2-diglycerides	Yes	(i) One-way ANOVA with Fisher´s test (ii) LDA (iii) Linear regression	SL estimated by linear regression using the 1st discriminant function as input variable	Di Serio et al. [49]

Table 1 (continued)

Olive oil sample	Storage conditions			Experimental assays		Statistical analysis	SL models	Reference
	Packaging material	Temperature	Time-period	Physical and Chemical parameters	Sensory analysis			
EVOO of different olive cultivars, degree of ripeness and geographical regions (Morocco, Portugal and Spain)	(i) PET bottles (60 mL) (ii) Light exposed during 12 h per day	Ambient temperature (20 ± 5 ºC)	14 months	PV, K_{232}, K_{270} Phenolic compounds Tocopherols Pyropheophytin Volatile compounds 1,2- and 1,3-diglycerides	Yes	(i) PCA (ii) PLS model	SL estimated using a PLS model based on the 17 selected parameters, with 4 latent variables SL or "critical time" (t_C) required the definition of acceptability limits for all parameters, even for those that are not legally regulated	Martin-Torres et al. [50]
Kinetic-empirical modelling strategies								
VOO of cv. Cornicabra	(i) Open glass bottles (ii) Dark conditions	20, 40, 50 and 60 ºC	19–93 weeks	PV, K_{232}, K_{270} Fatty acid profile Phenolic compounds Tocopherols	n.p	(i) Linear multiple regression models (MLRM) (ii) PCA (iv) One-way ANOVA with Tukey test	SL estimated by a fusion approach of pseudo first-order kinetic models with MLRM based on variables related to the oil's initial state and composition as well as to the oils' oxidation progress	Mancebo-Campos et al. [36]
Spanish EVOO of different cultivars, degree of ripeness and geographical regions	PET bottles	Ambient temperature (20ºC)	14 months	PV, K_{232}, K_{270} Phenolic compounds Tocopherols Pyropheophytin Volatile compounds 1,2- diglycerides	Yes	(i) PCA (ii) PLS model	SL estimated by using experimental data and the principal component scores (PC1, PC2, etc.) of PCA of the physicochemical-sensory dataset as input variables of pseudo zero-order, pseudo first-order, or pseudo second-order kinetic models	Martin-Torres et al. [50]

n.p.: information not provided

Kinetic Models

Conte et al. [37]

Conte et al. [37] studied Italian monovarietal olive oils (cv. Coratina) stored in sealed 250 mL transparent glass bottles, shielded from light, and subjected to different temperatures (25, 40, 50, and 60 ºC) for a duration of 300 days. Various parameters were analysed, including free acidity (FA, % oleic acid), peroxide value (PV, mEq O_2/kg oil), and specific extinction coefficients at 232 and 270 nm (K_{232} and K_{270}, respectively). Additionally, total phenolic compounds, tocopherols, pyropheophytin a, conjugated trienes, and volatile compounds were assessed. To depict the changes over time, pseudo zero-order kinetic models were applied to the experimental data of K_{232}, K_{270}, and pyropheophytin a. The impact of temperature on lipid oxidation was effectively described by the Arrhenius equation (Eq. (6), facilitating the determination of activation energies and frequency factors. The investigation revealed that the estimated shelf-life of the oils varied depending on the parameter under consideration, ranging from 32 to 377 days, with the shortest durations observed for K_{270} at 60 ºC and the longest for K_{270} at 25 ºC. Furthermore, the results indicated that peroxide value and antioxidant levels were inadequate for estimating the oil's shelf-life, whereas K_{270} emerged as the most reliable indicator parameter. Moreover, pyropheophytin a exhibited potential as a freshness index, demonstrating greater sensitivity to temperature fluctuations compared to the secondary oxidation index (K_{270}). On the other hand, the authors concluded that PV and antioxidant contents were not effective indicators of the SL of the studied olive oils.

Mancebo-Campos et al. [17]

Mancebo-Campos et al. [17] also studied monovarietal olive oils, cv. Cornicabra, some were collected directly from producers, and others were extracted using an Abencor pilot system. The oils (approximately 40 mL) were stored in open 125 mL amber glass bottles at different temperatures (25, 40, 50, and 60ºC) during different time periods (93, 41, 34, and 19 weeks, respectively), being periodically analysed. Based on an accelerated method, the study aimed to estimate the EVOO shelf-life based on the loss of its nutraceutical capacity and the inability to comply with nutritional and health claims approved by the EU regulations [38–41]. In fact, unlike other studies, the focus was not on the oil's SL in terms of commercial grade (i.e., EVOO, VOO, etc.) but, for the first time, on nutritional/health claims. The following claims were considered by the authors: (i) high unsaturated fat level, assessed from the mono- or polyunsaturated fatty acids contents and related to the benefits associated with the contribution to maintain or reduce the normal blood cholesterol levels by replacing saturated fats with unsaturated fats in the diet; (ii) source of $\omega-3$ fatty acids, assessed through the content of α-linolenic acid, and related with the known contribution to keep normal blood cholesterol levels, provided that a minimum daily intake of 2 g is ensured; (iii) source of vitamin E, which contributes the protection of cells from oxidative stress; and (iv) source of olive oil polyphenols, which contributes to the protection of blood lipids from oxidative stress, provided that the oil contains at least 5 mg of hydroxytyrosol and its derivatives per 20 g of olive oil, being required a minimum daily intake of 20 g of oil.

In this context, kinetic models, namely pseudo zero-order and pseudo first-order models [Eqs. (3) and (4)] were used, being the effect of temperature on the reaction rates described by the Arrhenius equation with or without the curvature correction [Eqs. (6) and (7)]. The study concluded that the evolution of the content of α-linolenic acid with the storage time followed a pseudo zero-order kinetic [Eq. (3)]. Contrary, for α-tocopherol (i.e., vitamin E) as well as for the sum of hydroxytyrosol, tyrosol, and their derivatives, a pseudo first-order kinetic [Eq. (4)] was the best model. For all parameters, a linear Arrhenius behaviour [Eq. (6)] was observed between the degradation reaction rate and the inverse of the storage temperature. The research also pointed out that, at each storage temperature, the degradation rate constants determined for each nutrient/compound depended on the oil sample under evaluation, whose maximum value was 1.6 to 2.6 times greater than the minimum value. Also, as expected, the degradation rate constant significantly increased with the storage temperature rise, being the major increase observed for α-tocopherol. The degradation rate constants were then used to calculate the claims' shelf-life, namely the TLHC (or TRLL), by applying Eqs. (8) and (9). Finally, according to the authors, the temperature dependence (with T in ºC) of the TLHC (i.e., SL in terms of the health claim) could be described by a power law such as:

$$TLHC = a \times T^{b} \tag{11}$$

where a and b are constants that can be calculated by linear regression of $ln\,(TLHC)$ versus $ln(T)$, using the data for all assays (different independent oil samples and storage temperatures).

The authors proposed for each compound/health claim studied, that the exponent b could be approximately constant for the temperature range evaluated (between 25 and 60 ºC), and thus Eq. (11) was be applied to predict the TLHC at ambient temperature (e.g., 25 ºC) from data obtained at higher temperatures (e.g., 40 ºC, 50 ºC or 60 ºC, ASLT) as follows:

$$a = \frac{TLHC_{T\neq 25^{\circ}C}}{\left(T_{\neq 25^{\circ}C}\right)^{b}} \tag{12}$$

$$TLHC_{25^{\circ}C} = a \times 25^{b} \quad (13)$$

However, for all the health claims considered, the results proven that the attempt to conduct a single test at a sole temperature between 40 and 60 ºC and using Eq. (12)-(13) to calculate the TLHC at 25 ºC was unfeasible due to the high discrepancy observed between the calculated and experimental TLHC values.

Calligaris et al. [19]

Calligaris et al. [19] investigated the shelf-life (SL) of extra virgin olive oil (EVOO) produced in Italy in 2019. The olive oils were categorized into three groups based on their initial total polyphenol concentrations (approximately 156, 273, and 507 mg/kg). The oils were produced immediately after harvesting and packaged within one month. Samples of 250 mL were stored under conditions mimicking commercial environments, using glass bottles with metal caps lined with polytetrafluoroethylene (PTFE) and with 2 cm of headspace. These samples were kept in incubators at controlled temperatures of 25, 40, 50, and 60 °C in the dark for up to 300 days, with sampling at predetermined intervals. To estimate SL, an accelerated shelf-life testing (ASLT) method was used, focusing on typical quality indicators such as PV, K_{232}, K_{270}, polyphenols, tocopherols, and pyropheophytins (%PPP). The study found that PV, K_{232}, polyphenols, and tocopherols did not significantly change under the evaluated storage conditions, never approaching legal limits for PV and K_{232}. This result was consistent with the findings of Conte et al. [37] but contrary to those reported by Mancebo-Campos et al. [36, 38]. The discrepancy was attributed primarily to different storage conditions, particularly closed versus open bottles, since in closed bottles, low oxygen availability in the headspace reduced the formation of primary oxidation products during storage. Conversely, significant changes were observed in K_{270} and %PPP, which were identified as the best indicators for monitoring oil behaviour during storage.

The SL estimation was conducted by assuming pseudo zero-order kinetics (Eq. 3) for both K_{270} and %PPP, which were proposed as the parameters that best allow monitoring the SL of olive oil during storage. The pyropheophytin index can be used as an early warning indicator of the product's performance on the market, and the K_{270} as a predictive indicator of SL. The apparent pseudo zero-order rate constants (k) were determined through linear regression analysis, with correlation coefficients ranging from 0.80 to 0.99. As anticipated, the rate constants (k) increased with rising temperature, independent of the initial total polyphenol concentration in the oils. However, the change in k values with temperature varied significantly depending on the initial total polyphenol concentration. For both K_{270} and %PPP, the temperature dependence of the k values was effectively described using a modified Arrhenius equation [Eq. (6)], with a reference temperature of 318 K, achieving correlation coefficients between 0.97 and 0.99:

$$k(T) = k_{ref} \times e^{\left[-\frac{E_a}{R}\left(\frac{1}{T}-\frac{1}{T_{ref}}\right)\right]} \quad (14)$$

In this way, the SL for a given temperature T (in Kelvin) could be estimated using Eq. (8) modified as:

$$SL = \frac{C_{limit} - C_0}{k_{ref} \times e^{\left[-\frac{E_a}{R}\left(\frac{1}{T}-\frac{1}{T_{ref}}\right)\right]}} \quad (15)$$

where C_{limit} was the limit of acceptability for the critical indicator.

Although the EU has not established legal thresholds for %PPP in EVOO, the authors evaluated this parameter as it has been suggested as an indicator of oil freshness [43–45]. Therefore, two SLs were estimated: the compulsory shelf-life (SL_C) based on K_{270} ($C_{limit} = 0.22$), and the high-quality shelf-life (SL_{HQ}) based on %PPP ($C_{limit} = 17\%$). In both cases, bootstrap confidence intervals were computed [46, 47].

Different SLs were obtained depending on the parameter evaluated, confirming the ASLT methodology's viability for determining EVOO's SL. The SL_C ranged from approximately 64 to 1161 days (mean values for the three EVOO groups), with the lowest value obtained for K_{270} at 60 ºC and the highest at 20 ºC. Particularly, mean SL_C values at 25ºC varied between 686 and 870 days, meeting the EU regulation requirement that olive oil should not exceed the acceptability limit before a SL of 18 months (540 days) at 25ºC. Since SL estimation relies on the initial value of K_{270}, monitoring this parameter (or others of interest) at the time of bottling is crucial. Regarding %PPP, the authors found that, although it is not an officially recognized quality indicator, it could serve as a "fast alert" signalling that compulsory limits could be reached soon due to its higher sensitivity to temperature changes compared to K_{270} (with higher activation energy values found for %PPP). Indeed, SL_{HQ} values ranged between 2 and 981 days (mean values considering the three EVOO groups), significantly lower than those determined based on K_{270} values. Finally, the authors concluded that the initial concentration of total polyphenols in freshly produced oils did not appear to be a critical factor in determining the temperature dependence of changes in K_{270} or %PPP, and thus the final SL value for EVOO stored in dark and low oxygen conditions.

Gagour et al. [48]

Gagour et al. [48] investigated the SL of olive oils from Arbequina and Moroccan Picholine cultivars, from Morrocco, extracted using a 3-phase extraction system. The olives, with a ripening index between 5 and 6, were harvested in 2020. To minimize oxidation, the oils were stored in sealed 30 mL glass bottles, protected from light, and kept in an oven at 60 ºC for 8 weeks. After this period, standard quality parameters and oxidative stability (OS) were assessed using the Rancimat method at temperatures ranging from 100 to 150 ºC. A thermodynamic approach was applied to estimate the SL. Instead of determining the rate constant (k) for each quality parameter at different storage temperatures by assuming a reaction order, the k values were calculated as the inverse of the OS (or induction time), measured at temperatures between 100 and 150 ºC using the Rancimat method:

$$k = \frac{1}{OS} \tag{16}$$

being k expressed in time^{-1} units, i.e., comparable to that of a pseudo first-order reaction.

As in other studies, the authors successfully described the temperature dependence of k using the Arrhenius equation (Eq. (6)), with satisfactory accuracy ($0.9946 \leq R^2 \leq 0.9970$). It was evident that the k values for both oils significantly decreased as the temperature increased. To estimate the SL of each monovarietal olive oil (cv. Arbequina or cv. Moroccan Picholine) at ambient temperature (25 ºC), the authors extrapolated the linear regression equations established between the accelerated temperature (T) of the Rancimat method and $ln(OS)$ for 298 K (25 ºC). This method, as the authors noted, is a preliminary but practical tool for predicting the SL of oils.

For Arbequina olive oils Gagour et al. [48] proposed the following equation:

$$T(K) = (-13.6 \pm 0.1) \times ln(OS, h) + 417.1 \pm 0.4 \tag{17}$$

and so, the shelf-life at 25 ºC was predicted by the researchers as,

$$SL_{25°C} = OS_{298K} = \frac{298 - 417.1}{-13.6} = 6357h \approx 9months$$

For Moroccan Picholine olive oils Gagour et al. [48] established the following:

$$T(K) = (-13.3 \pm 0.1) \times ln(OS, h) + 423.1 \pm 0.6 \tag{18}$$

and so, the shelf-life at 25 ºC was predicted by the researchers as,

$$SL_{25°C} = OS_{298K} = \frac{298 - 23.1}{-13.1} = 12161h \approx 17months$$

According to the SL predictions, Gagour et al. [48] found that the estimated SL at 25 ºC for Arbequina oils was significantly lower than that of Moroccan Picholine oils, indicating a significant effect of the cultivar. However, it is important to recognize that predicted values based on extrapolation procedures may be susceptible to errors.

Empirical Models

Di Serio et al. [49]

The studied EVOO were extracted using a 3-phase continuous centrifugation system from handpicked olives of different cultivars, namely cvs. Biancolilla, Carolea, Coratina, Dolce di rossano, Frantoio, Nocellara del Belice and Nocellara Etnea. The oils were stored in closed 1 L dark green bottles, exposed to diffuse light at room temperature, with mean temperatures of 15 ºC in winter and 18 ºC in summer. These EVOOs were evaluated at bottling (0 months) and at 2-month intervals up to 12 months. The analyses included: free acidity (FA), peroxide value (PV), UV spectrophotometric indices, ethyl esters, 1,2-diglycerides, tocopherols, phenolic compounds, volatile compounds, fatty-acid composition, sterol composition, and sensory profile.

The experimental data gathered during the study was further used to develop a mathematical model, using an empirical approach, that allowed relating the age of the oil (Y_{age}) and the discriminant functions (Yd) by means of a single linear equation:

$$Y_{age} = a \times Y_d + b \tag{19}$$

The discriminant function Y_d was computed by means of the weighted linear combination of the independent variables selected by the stepwise heuristic algorithm. For the analysis, Di Serio et al. [49] split the EVOO samples into seven groups according to the storage time (0, 2, 4, 6, 8, 10 and 12 months), which included one independent oil from each of the seven cultivars studied. The LDA showed that the 1st discriminant function allowed explaining a great percentage of the original data variability, and thus, the authors proposed it as the Y_d function.

$$\begin{aligned} Y_d = {} & 0.0621 \times \left[alkylesters\left(mgkg^{-1}\right)\right] + 0.268 \\ & \times \left[1{,}2 - diglicerides(\%)\right] - 0.347 \times \left[GlobalQualityIndex\right] \\ & -0.0672 \times \left[Oxidized - phenols\right]\left(mgkg^{-1}\right) - 0.0298 \\ & \times \left[trans - 2 - hexenal \times \frac{100}{totalvolatilecompounds}(\%)\right] - 16.458 \end{aligned} \tag{20}$$

Substituting Eq. (20) into Eq. (19), together with the values of a and b, obtained by linear regression analysis using the groups' centroids ($a = -1.051$ and $b = +6.000$), the following equation was proposed by Di Serio et al. [49] for estimating the oils' age:

$$\begin{aligned} Y_{age} &= 0.0653 \times \left[alkylesters\left(mgkg^{-1}\right)\right] - 0.282 \\ &\times \left[1{,}2 - diglicerides(\%)\right] + 0.364 \times \left[GlobalQualityIndex\right] \\ &+ 0.0706 \times \left[Oxidized - phenols\right]\left(mgkg^{-1}\right) + 0.0313 \\ &\times \left[trans - 2 - hexenal \times \frac{100}{totalvolatilecompounds}(\%)\right] + 23.297 \end{aligned} \tag{21}$$

Di Serio et al. [49] demonstrated that Eq. (21) could estimate the age of unknown EVOOs with an accuracy of ± 1 month. This allows for determining the remaining storage time or SL of the oil, assuming a typical SL of about 11 months for EVOO. However, it is important to note that the accuracy of this oil age model relies on various physicochemical experimental data. This requirement makes the procedure time-consuming and expensive, rendering it not viable for most small olive oil producers.

Martin-Torres et al. [50]

Martin-Torres et al. [50] evaluated the physicochemical changes suffered by 83 commercial olive oils, from different cultivars, degrees of ripeness and geographical regions (Morocco, Portugal and mainly Spain) when stored in closed 60 mL PET bottles at room temperature (20ºC ± 5ºC) exposed during 12 h to a cool white LED light for 14 months. In this sense, the authors followed an actual shelf-life testing, mimicking the expected real storage conditions carefully chosen to realistically simulate storage. Olive oils were analysed at 0 months and then consecutively every two months. Different parameters were evaluated namely, K_{232}, K_{270} and ΔK, oxidative stability (Rancimat method), peroxide and anisidine values, 1,2- and 1,3-diglycerides, tocopherols, pyropheophytin a, phenolic compounds (e.g., phenolic alcohols, phenolic acids, secoiridoids, lignans and flavonoids relative abundance), volatile compounds, as well as the intensity of rancid. An empiric approach based on unsupervised qualitative (Principal Component Analysis, PCA) and quantitative (Partial Least-Squares, PLS) multivariate statistical techniques was applied aiming to estimate the SL (i.e., oils age). For that, first Martin-Torres et al. [50] assessed the eventual existence of outliers by PCA, concluding that all variables and samples could be included in the study. Then, a preliminary PLS model was developed by the researchers that allowed establishing a relationship between storage time (dependent variable) and the significant dependent physicochemical-sensory parameters (independent variables). The modelling strategy identified 17 significant parameters (PV, K_{232}, K_{270}, ΔK, OS, rancidity, phenolic alcohols, α- and β- tocopherol, 1,2-diglycerides, 7 volatiles among the 9 detected, and pyropheophytin a) out of initial 30 variables studied. A PLS model based on the 17 selected parameters was developed, comprising 4 latent variables (LV1 to LV4). The model allowed calculating the storage time of the studied oils ($R^2 = 0.94$, RMSE = 0.004). Moreover, based on LV1 scores, a linear model was established between the autoscaled experimental values and the storage time ($R^2 = 0.91$, RMSE = 0.89).

However, to estimate the SL also called the "critical time" (t_C), the authors needed to define acceptability limits for all the parameters included in the PLS model, even for those that are not legally regulated, which, although based on some experimental evidence, may pose some reliability issues.

Finally, the following equation was used for calculation:

$$Q_c = max(\boldsymbol{Q_a}.\boldsymbol{L}) \tag{22}$$

with $\boldsymbol{Q_a}$ as the autoscaled acceptability limit vector, and $\boldsymbol{L}$ as the loading vector of LV1.

The t_C value, i.e., the maximum ageing time at which an olive oil cannot be classified as EVOO anymore (cut-off criteria or SL), was then obtained after interpolating QC into the SL model equation. Once the model has been established and t_C has been estimated, the equivalent ageing time t_i of each olive oil sample was calculated by the researchers by applying the following equation: ti can be calculated using the following formula:

$$Q_i = \boldsymbol{X_i}.L \rightarrow Q_i = -4.267 + 0.60096 \times t_i \tag{23}$$

being $\boldsymbol{X_i}$ the autoscaled vector of experimental data for each sample. Q_i is interpolated into the SL model to obtain t_i.

Based on these values, the authors proposed a SL index (I_{SL}) that corresponds to the number of months for which an EVOO still complies with the requirements of its category, being calculated by:

$$I_{SL} = t_c - t_i \tag{24}$$

with t_c set equal to 13 months.

The ageing rate of the EVOO can then, according to Martin-Torres et al. [50], be calculated as:

$$\%Age = \frac{t_i}{t_c} \text{x } 100 \tag{25}$$

The authors successfully validated the proposed empirical approach by applying it to estimate the SL of 5 EVOO samples randomly selected from those included in the study, being pointed out that, according to the authors' opinion, the methodology could be accurately applied to oils stored under different conditions, like different bottling materials. Nevertheless, it must be highlighted that the proposed methodology is mathematically complex and requires the input of

a significant number of experimental data, which may render its application a difficult and expensive task.

Kinetic-Empirical Merged Models

Mancebo-Campos et al. [36]

Mancebo-Campos et al. [36] studied EVOO from cv. Cornicabra extracted in both industrial mills and using an Abencor pilot system. The oils were stored in open 125 mL amber glass bottles, in the dark, at different temperatures (25, 40, 50 and 60ºC) for 650 days. The composition of the olive oils was monitored during the storage time-period. In a previous study of the same research team [42], the following parameters were assessed: PV, K_{232} and K_{270}, $C_{18:1}$, $C_{18:2}$ and $C_{18:3}$ fatty acids, total phenols calculated as the sum of hydroxytyrosol, tyrosol and their derivatives, α-tocopherol and the oxidative stability (OS, Rancimat). In the study of 2022, the authors monitored additional parameters of the same olive oils, including the contents of chlorophyl, carotenoids, individual phenols, and o-diphenols. Pseudo first-order kinetic models (Eq. (3)) were proposed by Mancebo-Campos et al. [36] to describe the evolution of secoiridoids hydroxytyrosol, tyrosol, total phenols and o-diphenols ($0.739 \le R^2 \le 0.997$), being observed, for each parameter, an increasing trend of the reaction rate constants (k) with the temperature, which temperature dependence was satisfactorily fitted by the Arrhenius equation (Eq. (6), $0.862 \le R^2 \le 0.998$). Contrary, hydroxytyrosol, tyrosol and α-tocopherol contents were fitted using pseudo zero-order kinetic models ($0.425 \le R^2 \le 1.000$) and the reaction rate constants (k) also increased with the temperature, being this behaviour successfully described by the Arrhenius Eq. ($0.524 \le R^2 \le 0.992$). The different E_a values calculated by fitting the Arrhenius equation showed that the studied parameters had different temperature susceptibility. On the other hand, according to Mancebo-Campos et al. [36, 42], no marked relationship could be established between the E_a values of the oxidation reactions and the OS or the oils' SL (TRUL model). In fact, as discussed by the authors, the values of E_a and k should be used from a mechanistic point of view to interpret the observed degradation reactions since oxidation involves a complex and broader set of reactions, although they may be used as descriptive tools of the temperature dependence of the oxidation reaction.

To estimate the SL of the studied oils, Mancebo-Campos et al. [36] proposed a combined kinetic-empirical approach based on 16 variables related to the oils' initial state and composition, including contents of carotenoids, chlorophylls, hydroxytyrosol and tyrosol and their respective secoiridoids, total phenols, o-diphenols, α-tocopherol, unsaturated and polyunsaturated fatty acids, PV, extinction coefficients, and Rancimat oxidative stability. Additionally, they considered 13 variables related to the oils' oxidation progress, such as rate constants of PV, extinction coefficients, total phenols, o-diphenols, hydroxytyrosol secoiridoids, α-tocopherol, TRUL values of PV and extinction coefficients, and experimental oxidation temperatures. The authors attempted to develop MLRM using these variables, and two additional ones associated with the health claim related to the contents of olive oil polyphenols: the decreasing rates of o-diphenols (hydroxytyrosol and derivatives) and hydroxytyrosol secoiridoids, both measured at 25 ºC. They established different MLRMs based on a reduced number of non-redundant independent variables selected by a stepwise heuristic algorithm. Although some models exhibited high R^2 values (> 0.937), the authors concluded that these models had limited predictive value due to the complexity of the calculations involved to used experimental factors. As an alternative, Mancebo-Campos et al. [36] proposed MLRMs based on more experimentally accessible variables, specifically the initial composition data and degradation rates calculated for accelerated storage temperatures (40 and 50 ºC), achieving R^2 values between 0.881 and 0.915. However, it should be noted that all the developed models involved a significant mathematical complexity, making their practical implementation challenging for olive oil producers.

Martin-Torres et al. [50]

Martin-Torres et al. [50] attempted to apply a kinetic-empirical merged model to estimate the SL of EVOO from various geographical origins. The oils were stored in 60 mL PET bottles at 20 ± 5 ºC and exposed to cool white LED light for 12 h a day over a period of 14 months (as described in Sect. "Martin-Torres et al. [50]"). The authors proposed fitting the experimental data and the principal component scores (PC1, PC2, etc.) derived from the PCA of the physicochemical-sensory dataset as input variables to establish kinetic models of pseudo zero-order, pseudo first-order, or pseudo second-order (Eqs. (3) to (5)). They also evaluated the Weibull kinetics. The study concluded that the best model fitting was achieved using a pseudo first-order kinetic degradation equation for the PC1 scores. However, even in this case, the R^2 value was relatively low ($R^2 = 0.894$; RMSE = 0.0489), leading the authors to decide that the merged approach was not promising for shelf-life estimation.

Brief Overview of Empirical and Kinetic Approaches for SL Estimation

As pointed out previously, two primary modelling approaches are described in the literature for estimating and predicting the shelf-life (SL) of olive oils: empirical-based and kinetic-based approaches. The kinetic-based approach is more widely used than the empirical-based approach.

The empirical approach relies on a comprehensive set of physicochemical and sensory parameters, which, in theory, can provide more robust and potentially realistic SL estimations. However, a critical overview of the existing literature reveals some limitations and drawbacks associated with this strategy:

- Empirical models require a larger experimental dataset, often encompassing information on the chemical composition of the oils, such as phenolic compounds, tocopherols, volatile compounds, and the intensities of sensory sensations. While this data is valuable, it is not legally required, making this approach time-consuming and costly. Moreover, its implementation is typically beyond the technical and financial capabilities of small and medium-sized olive oil producers.
- Empirical models, developed using multivariate statistical techniques, often result in complex equations that are neither easy to implement nor straightforward for small and medium-sized olive oil producers to use in practice as a routine tool for SL estimation.
- Lastly, since empirical models are typically developed using a large experimental dataset that not only includes the legally defined quality parameters for grading olive oils (such as EVOO, VOO, or LOO), or for labelling oils with regulated nutritional and health claims, establishing acceptability limits for SL estimation becomes necessary. These limits, while generally proposed based on experimental evidence, lack legal support. This lack of regulatory support can hinder the commercial application of findings derived from these empirical approaches.

On the other hand, kinetic-based models are typically designed using parameters for which legal thresholds exist for oils. However, these models rely on a single-parameter approach and are thus significantly influenced by the oil's composition during bottling. This composition is affected by factors such as the crop year, harvest time and conditions, olive cultivar, and several external influences during storage, including the type of packaging material and storage conditions (e.g., temperature and exposure to light or darkness). However, the mathematical simplicity of the SL prediction models, the limited number of experimental parameters needed, and the existence of legal thresholds make kinetic-based approaches more promising for SL estimation.

In the next section, a comprehensive overview of the kinetic-based approaches reported in the literature is provided. This includes a review of the most recent papers, as well as those published before 2018, which were covered in a previous review by Li and Wang [22]. The primary goal is to critically evaluate the findings related to degradation kinetic rate constants and SL estimations based on the key quality parameters most used for modelling purposes: peroxide value, and extinction coefficients. When the original papers did not provide these data, the experimental data published by the authors were used to estimate the *k* and/or SL values, employing the most used kinetic models (i.e., pseudo zero-order and pseudo first-order equations). The original and/or calculated values were then converted to the same units as needed to facilitate comparisons. Studies focusing on SL estimation based on nutritional and/or health claims were excluded due to the limited number of papers on this topic, which precluded a comparative evaluation. Ultimately, the aim is to determine which of the considered quality parameters provides a more conservative SL prediction, meaning the one most sensitive to storage conditions and thus seems to be the most promising SL indicator.

Critical Assessment of the Kinetic Models Reported in the Literature for SL Estimation

Tables 2, 3, and 4 summarize the findings from various studies on estimating the SL of olive oils using kinetic-based models developed by different researchers. These tables focus on three quality parameters: *PV*, K_{232}, and K_{270}. Indeed, the literature indicates that while FA is a legally recognized critical quality parameter for determining the commercial quality of olive oils, it is not a suitable SL indicator. This is primarily because FA remains stable during storage under various conditions. The data spans studies published between 2000 and 2023. To create a more comprehensive dataset, when the original studies did not provide the kinetic rate constants (*k*) and SL values, these were calculated in the present study using the original experimental data. The data were fitted using either pseudo zero-order or pseudo first-order kinetic equations, with the best-fitting values reported. For ease of comparison, all parameters are presented in the same units.

The analysis in this study demonstrated that regardless of the parameters used to develop the SL predictive models, pseudo zero-order and pseudo first-order kinetic equations were the most employed. Additionally, among the three quality parameters, the extinction coefficient K_{270} was most frequently used for developing kinetic models to estimate the SL of olive oils. This was followed by the extinction coefficient K_{232}, and lastly, by *PV*. This preference may be attributed to the fact that most studies were conducted with fully closed bottles, which had limited headspace and oxygen availability. This environment restricts primary oxidation reactions, thereby limiting the increase in *PV* and K_{232}, while promoting secondary oxidation reactions that affect K_{270} levels. The data presented in Tables 2, 3, and 4 show that at lower storage temperatures (20–25 ºC), kinetic models based on K_{232}, K_{270}, or *PV* predicted similar average SLs for oils stored in closed bottles: 351, 478, and 392 days, respectively.

Table 2 Kinetic models reported in the literature for estimating the olive oils shelf-life (SL) based on the evolution of the peroxide value (*PV* in mEq O_2/kg of oil) during storage: olive oil information (geographical origin, olive cultivars), storage conditions (packaging material, storage time, storage temperature), pseudo order of the kinetic model (n), mean oxidation rate constant ± standard deviation ($k \pm$ sd), SL, and literature reference

Geographical origin	Olive cultivar	Packaging material	Storage time (months)	Kinetic model				References
				Storage temperature (ºC)	n	$k \pm$ sd (mEq. O_2 kg^{-1} day^{-1})	SL (days)	
Spain	Cornicabra	Amber glass bottles (open)	23	25	0	$(0.19 \pm 0.04) \times 10^{-1}$	853 ± 180	[42]
Portugal	Cobrançosa, Verdeal Transmontana and Madural	Dark amber glass bottles	12	25	0	0.44×10^{-1}	392	[51]#
Spain	Cornicabra	Amber glass bottles (open)	23	40	0	$(0.79 \pm 0.24) \times 10^{-1}$	190 ± 48	[42]
Spain	Cornicabra	Amber glass bottles (open)	23	50	0	$(1.6 \pm 0.4) \times 10^{-1}$	87 ± 17	[42]
Not referred	Not referred	Glass bottles	Not referred	50	0	$(4.1 \pm 2.1) \times 10^{-1}$	26 ± 15	[52]#
Morocco	Arbequina	Not referred	2	60	0	0.70	24	[48]#
Morocco	Moroccan Picholine	Not referred	2	60	0	0.39	45	[48]#
Spain	Cornicabra	Amber glass bottles (open)	23	60	0	$(3.1 \pm 1.0) \times 10^{-1}$	44 ± 12	[42]

k and SL values were calculated in this study using the original data reported by each study

Table 3 Kinetic models reported in the literature for estimating the olive oils shelf-life (SL) based on the evolution of the UV–Vis extinction coefficient at 232 nm (K_{232}) during storage: olive oil information (geographical origin, olive cultivars), storage conditions (packaging material, storage time, storage temperature), pseudo order of the kinetic model (n), mean oxidation rate constant ± standard deviation ($k \pm$ sd), SL, and literature reference

Geographical origin	Olive cultivar	Packaging material	Storage time (months)	Kinetic model				References
				Storage temperature (ºC)	n	$k \pm$ sd (day^{-1})	SL (days)	
Italy	Canina, Frantoiano, Moraiolo, and Leccino	Dark glass bottles	21	20	0	0.20×10^{-2}	413	[30]#
Spain	Cornicabra	Amber glass bottles (open)	23	25	0	$(0.19 \pm 0.06) \times 10^{-2}$	431 ± 162	[42]
Portugal	Cobrançosa, Verdeal Transmontana, and Madural	Dark amber glass bottles	12	25 (Light)	0	0.30×10^{-2}	409	[51]#
Portugal	Cobrançosa, Verdeal Transmontana, and Madural	Dark amber glass bottles	12	25 (Dark)	0	0.53×10^{-2}	230	[51]#
Spain	Arbequina	Amber glass bottles (open)	23	40	0	$(1.04 \pm 0.23) \times 10^{-2}$	71 ± 21	[42]
Spain	Cornicabra	Amber glass bottles (open)	23	50	0	$(2.16 \pm 0.51) \times 10^{-2}$	31 ± 10	[42]
Morocco	Arbequina	Not referred	2	60	1	0.77×10^{-2}	23	[48]#
Morocco	Moroccan Picholine	Not referred	2	60	1	0.93×10^{-2}	41	[48]#
Spain	Cornicabra	Amber glass bottles (open)	23	60	0	$(3.74 \pm 0.95) \times 10^{-2}$	18 ± 6	[42]

k and SL values were calculated in this study using the original data reported by each study

Table 4 Kinetic models reported in the literature for estimating the olive oils shelf-life (SL) based on the evolution of the UV–Vis extinction coefficient at 270 nm (K_{270}) during storage: olive oil information (geographical origin, olive cultivars), storage conditions (packaging material, storage time, storage temperature), pseudo order of the kinetic model (n), mean oxidation rate constant ± standard deviation ($k \pm$ sd), SL, and literature reference

Geographical origin	Olive cultivar	Packaging material	Storage time (months)	Kinetic model				References
				Storage temperature (ºC)	n	$k \pm$ sd (day^{-1})	SL (days)	
Italy	Not referred	Clear glass bottles	10	25	0	$(0.18 \pm 0.01) \times 10^{-3}$	377	[37]
Italy	Not referred	Not referred	10	25	0	$(0.13 \pm 0.01) \times 10^{-3}$	873 ± 76	[19]
Spain	Cornicabra	Amber glass bottles (open)	23	25	1	$(0.87 \pm 0.19) \times 10^{-3}$	548 ± 157	[42]
Portugal	Cobrançosa, Verdeal Transmontana, and Madural	Dark amber glass bottles	12	25 (Light)	0	2.17×10^{-3}	185	[51]#
Italy	Not referred	Clear glass bottles	10	40	0	$(0.62 \pm 0.02) \times 10^{-3}$	122	[37]
Italy	Not referred	Not referred	10	40	0	$(0.37 \pm 0.09) \times 10^{-3}$	316 ± 94	[19]
Spain	Cornicabra	Amber glass bottles (open)	23	40	1	$(6.28 \pm 0.75) \times 10^{-3}$	112 ± 32	[42]
Italy	Not referred	Clear glass bottles	10	50	0	$(1.07 \pm 0.07) \times 10^{-3}$	61	[37]
Italy	Not referred	Not referred	10	50	0	$(0.96 \pm 0.22) \times 10^{-3}$	119 ± 25	[19]
Spain	Cornicabra	Amber glass bottles (open)	23	50	1	$(13.4 \pm 2.6) \times 10^{-3}$	38 ± 13	[42]
Italy	Not referred	Clear glass bottles	10	60	0	$(2.2 \pm 0.02) \times 10^{-3}$	32	[37]
Spain	Cornicabra	Amber glass bottles (open)	23	60	1	$(22.9 \pm 2.8) \times 10^{-3}$	19 ± 7	[42]
Italy	Not referred	Not referred	10	60	0	$(1.49 \pm 0.41) \times 10^{-3}$	78 ± 24	[19]
Morocco	Arbequina	Not referred	2	60	1	15.1×10^{-3}	35	[48]#
Morocco	Moroccan Picholine	Not referred	2	60	1	19.1×10^{-3}	17	[48]#

k and SL values were calculated in this study using the original data reported by each study

However, these predicted SLs fall short of the EU regulation requirement of a minimum 18-month (540 days) SL at 25 ºC. Surprisingly, models for oils stored in open bottles at 25 ºC indicated higher SL values (853, 431, and 548 days for PV, K_{232} and K_{270}, respectively), despite the expectation of higher oxidation rates due to greater oxygen availability. Particularly, SL drastically decreases at storage temperatures of 40 ºC or higher, which is crucial for olive oils destined for export. Major olive oil producers in the Mediterranean region export their products globally, and during intercontinental transportation, especially if not refrigerated, the oils' containers may reach high temperatures. Studies indicate that at 40–50 ºC, the SL of olive oil can decrease from over 350 days at 25 ºC to less than 200 days, posing significant challenges for exporters. The variability in SL values among studies at the same storage temperature can be attributed to several factors, including olive cultivar, geographical origin, extraction process (pilot versus industrial, and two-phase versus three-phase systems), packaging material, and exposure to light. Additionally, the initial composition of the oils at bottling, particularly the initial content of minor bioactive compounds like polyphenols and tocopherols, which contribute to the oil's antioxidant activity, plays a crucial role in SL estimation, especially for parameters with established legal thresholds. A detailed analysis of the gathered information (Tables 2, 3, and 4) indicates that at a fixed temperature and kinetic pseudo order reaction, the rate constants (k values) show minimal variability and are thus less affected by the former mentioned factors. For instance, at 25 ºC, the pseudo zero-order reaction rate constants found by different researchers varied slightly: 0.19×10^{-1} to 0.44×10^{-1} mEq O_2 kg^{-1} day^{-1} for PV, 0.19×10^{-2} to 0.30×10^{-3} day^{-1} for K_{232}, and 0.13×10^{-3} to 0.18×10^{-3} day^{-1} for K_{270}. A similar situation could be observed for the pseudo zero-order reaction rate constants at, for example, 40 ºC: 1.04×10^{-2} to 2.16×10^{-3} day^{-1} for K_{232}, and 0.37×10^{-3} to 1.07×10^{-3} day^{-1} for K_{270}. These findings confirm that differences in estimated SL are largely due to the initial physicochemical composition of the oils. Thus, it could be tentatively proposed the use of average k values at a specific storage temperature, to predict SLs of new oils if the initial values of PV, K_{232}, or K_{270} at bottling are known:

$$k_{PV,25°C} = 0.32 \times 10^{-1} mEq.O_2 kg^{-1} day^{-1}$$

$$k_{PV,40-50°C} = 2.16 \times 10^{-1} mEq.O_2 kg^{-1} day^{-1}$$

$$k_{K_{232},25°C} = 0.23 \times 10^{-2} day^{-1}$$

$$k_{K_{232},40-50°C} = 1.60 \times 10^{-2} day^{-1}$$

$$k_{K_{270},25°C} = 0.23 \times 10^{-3} day^{-1}$$

$$k_{K_{270},40°C} = 0.76 \times 10^{-3} day^{-1}$$

This hypothesis needs to be validated and refined with future data to enhance its predictive potential and facilitate its implementation as a routine tool in the olive oil field.

Conclusions and Future Perspectives

Estimating the shelf-life of olive oil is crucial for its commercial success and can significantly boost consumer confidence in this premium product. The literature identifies several methods for determining shelf-life, primarily categorized into kinetic-based models, empirical-based models, or a combination of both. Kinetic models focus on the changes in a single quality or composition parameter over time under specific storage conditions, such as temperature, packaging, and light exposure. In contrast, empirical models use a broader set of parameters, requiring advanced multivariate statistical techniques. While these empirical models can be highly accurate, their complexity often makes them impractical for non-experts, limiting their widespread use in the olive oil industry. Additionally, empirical models often rely on chemical data for compounds without established legal thresholds, necessitating arbitrary acceptability limits that lack formal support. Moreover, utilizing a larger database involves greater experimental and technical effort, making the approach highly expensive in time and money. Kinetic models, however, use simpler mathematical approaches that are easier to implement by non-specialists. These models apply various reaction orders (e.g., pseudo zero-, first-, or second-order equations) to describe changes in key quality parameters like peroxide value, extinction coefficients, and concentrations of bioactive compounds such as tocopherols and polyphenols. Generally, kinetic shelf-life models are categorized based on the time required for parameters to reach either upper legal limits (for quality parameters) or lower legal limits (for bioactive compounds). Most research has focused on developing models to estimate the shelf-life based on the commercial quality of olive oils, specifically the duration an olive oil remains within the legal standards for "extra virgin" or "virgin" labels. There is, however, a significant gap in models predicting shelf-life in terms of nutritional or health claims despite their high commercial value and consumer importance. Thus, collaboration between academia and the olive oil industry is essential to address this gap, enhancing the routine use of nutritional and health claims on labels. Among the various storage conditions, temperature has been identified as the most influential factor affecting olive oil shelf-life. However, more research is needed on the impact of packaging materials, including active and intelligent packaging, and the initial quality of olive oil at bottling on its estimated shelf-life. In conclusion, although this field remains a hot area of research, it is imperative for academia and industry partners to collaborate on developing simple, cost-effective, and accurate shelf-life predictive models. Such models would not only enhance consumer confidence but also establish definitive storage and transportation guidelines to maintain the high quality of olive oil.

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Data Availability No datasets were generated or analysed during the current study.

Declarations

Conflict of Interest The authors declare no competing interests.

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