

Shelf Life of Olive Oil and Useful Methods for its Prediction

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This review of the scientific literature examines the most relevant shelf-life indicators and models that are useful for predicting the shelf life of extra virgin olive oil. Keywords such as “olive oil”, “shelf life”, “freshness”, “best before date”, “kinetic model” and “prediction” were used using electronic journal databases.

BACKGROUND

“Shelf life” is most commonly defined as a length of time during which a food product retains a required level of quality under well-defined storage conditions (1). Consumers rely on shelf-life determinations to differentiate between food products that are acceptable for consumption from those that are no longer acceptable. Proper packaging and storage conditions can help to maintain the integrity of a product and maximize shelf life.

The most significant factor affecting the shelf life of olive oil is oxidation. Oxidation occurs when unsaturated fatty acids decompose to form odorless and tasteless hydroperoxides, which then degrade into compounds that are responsible for rancid flavors. Oxidation also reduces healthful phenols in olive oil and can lower the quality grade of the oil (2-4). The rate of olive oil oxidation varies depending upon the oil’s chemical composition (such as the levels of phenols, tocopherols and fatty acids profile), as well as the oil’s exposure to heat, light and oxygen in bulk storage and packaging.

In literature, the most widely applied chemical quality parameters for olive oil shelf life are free fatty acidity (FFA); peroxide value (PV); ultraviolet absorbance (UV); 1, 2-diacylglycerols (DAGs); pyropheophytins (PPP); sensory; induction time; total phenols and volatiles (Table 1). All but the last three of these parameters have established limits within quality standards from the International Olive Council (IOC) (2), United States Department of Agriculture (USDA) (3) and/or California Department of Food and Agriculture (CDFA) (4).

Table 1. Critical quality parameters for olive oil shelf life.

PARAMETER	DETERMINATION	INDICATOR	METHODOLOGY	CA EVOO STANDARD
Free Fatty Acids (FFA)	Free fatty acids are formed by the hydrolysis of the triacylglycerols during extraction, processing and storage.	An elevated level of free fatty acid indicates hydrolyzed fruits and/or poor quality oil made from unsound fruit, improperly processed or stored oil.	Analytical Titration	≤ 0.5 % as oleic acid
Peroxide Value (PV)	Peroxides are primary oxidation products that are formed when oils are exposed to oxygen, producing undesirable flavors and odors.	An elevated level of peroxides indicates oxidized and/or poor quality oil.	Analytical Titration	≤ 15 meq O ₂ /kg oil
Ultraviolet Absorbance (UV)	Conjugated double bonds are formed from natural nonconjugated unsaturation in oils upon oxidation. The K ₂₃₂ measures primary oxidation	An elevated level of UV absorbance indicates oxidized and/or poor quality oil.	UV spectrophotometry	K ₂₃₂ : ≤ 2.40 K ^{1%} _{1cm} ; K ₂₇₀ ≤ 0.22 K ^{1%} _{1cm} ; ΔK: ≤ 0.01 K ^{1%} _{1cm}

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	products and K_{270} measures secondary oxidation products.			
1, 2-Diacylglycerols (DAGs)	Fresh extra virgin olive oil contains a high proportion of 1,2-diacylglycerols to 1,2- and 1,3-diacylglycerols, while olive oil from poor quality fruits and refined olive oils have higher level of 1,3-DAGs than fresh extra virgin olive oils.	The ratio of 1,2-diacylglycerols to 1,2- and 1,3-diacylglycerols is an indicator for oil that is hydrolyzed, oxidized, and/or of poor quality.	Gas Chromatography (GC)	$\geq 35\%$
Pyropheophytins (PPP)	Chlorophyll pigments break down to pheophytins and then pyropheophytins upon thermal degradation of olive oil.	An elevated level of pyropheophytins is an indicator for oil that is oxidized and/or adulterated with refined oil.	High performance liquid chromatography (HPLC)	$\leq 17\%$
Sensory	Sensory refers to taste, odor and mouthfeel	Sensory assessment can help identify oils that are of poor quality, oxidized, and/or adulterated with other oils.	IOC-recognized panel of 8-12 people evaluates oils for sensory characteristics.	Median of defects=0.0; median of the fruity>0.0
Induction Time	The aging process is accelerated by means of heating up the reaction vessel and by passing air continuously through the sample.	Oxidative stability (in hours) denotes the resistance of oils to oxidation. The longer the induction time, the more stable the sample is.	Rancimat	NA
Total Phenols	The sum of up to 30 individual phenols, which are antioxidants that slow down oxidation.	A low level of total phenols can indicate a shorter shelf life while a high level of total phenols can indicate a longer shelf life.	UV spectrophotometry/ High performance liquid chromatography HPLC	NA
Volatiles (e.g. hexanal/nonanal, E-2-hexenal/hexanal),	Volatile compositions change during oxidation. For example, as the oil oxidizes, the concentration of hexanal decreases as concentration of nonanal increases.	The ratios of hexanal/nonanal and E-2-hexenal/hexanal can indicate oxidized oil.	Headspace-Gas Chromatography (GC)	NA

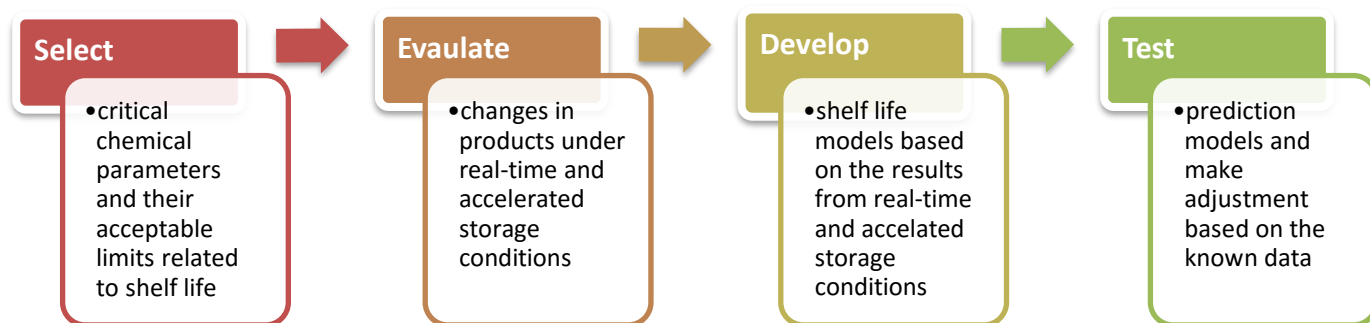
Published studies that examine the shelf life of extra virgin olive oil have mostly focused on the influence of packaging and/or storage conditions (5-7). Based on our review of these studies, we have identified best practices to minimize oxidation as summarized in Table 2.

Table 2. Best practices on packaging and storage conditions for olive oil shelf life.

PACKAGING	TEMPERATURE	LIGHT
Dark glass, aluminum cans with food-grade enamel coating, coated paperboard, and bag-in-box provide protection from light and oxygen. Bag-in-box has the advantage of maintaining minimum oxygen in headspace (8).	Stored at a reduced temperature of 15 °C (59 °F) (9).	Stored in the dark to minimize light exposure.

SHELF-LIFE PREDICTION MODEL DEVELOPMENT PROCESS

A typical shelf-life study starts with the research team selecting chemical parameters that are related to a product's shelf life – examining the changes that occur in each chemical parameter over time and determining an acceptable limit for each parameter. Next the team records the change in each parameter under real-time and accelerated storage conditions. Accelerated storage conditions seek to estimate temporal changes in the food by exposing the food to excessive temperature, oxygen or light. While this approach provides a more rapid and less-expensive method of predicting shelf life than monitoring a product in real time under normal storage conditions, some accelerated conditions may provide erroneous shelf-life predictions due to different chemical reaction mechanisms from the real-time conditions (10). Thus, shelf-life prediction models are best developed based on results from both real-time and accelerated storage conditions, followed by extensive evaluation and adjustment (Figure 1).

**Figure 1. Shelf life prediction method development process.**

POTENTIAL SHELF-LIFE PREDICTION MODELS FOR EXTRA VIRGIN OLIVE OIL

In general, there are two types of prediction models - kinetic models and empirical models:

- Kinetic models** are developed based on how reaction rates in individual chemical parameters (e.g., FFA, UV, PV, DAGs and PPP) are influenced by experimental conditions related to variables such as time, temperature and light. Data describing the changes of these parameters under conditions simulating actual storage are submitted to modeling based on the known rate of a particular reaction. The limitation of kinetic modeling is that classical kinetic equations cannot easily accommodate the complexity of oxidation reactions and olive oil deteriorations.
- Empirical models** are developed based on the *correlations* between individual parameters as a function of different variables. In this case, the fundamental kinetic analysis can be skipped and advanced statistical software is often used to perform linear regression and the best-fitting analysis. This enables a model to

demonstrate the relations between variables (e.g., FFA, UV, PV, DAGs and PPP) and a response variable (e.g., time in storage) by fitting a linear equation to observed data. The limitation of empirical modeling is the difficulty to extend beyond the measured setup (e.g., storage condition) and simplification and approximation can fail when the setup changes.

Although research efforts on shelf-life and best-before-date estimation have been made in fresh and some processed foods, most of the studies on extra virgin olive oil did not propose a clear shelf-life prediction model (11-16). However, there were four studies that did propose useful models which are discussed in the following pages in the order of ease of adoption and readiness to be utilized.

1. **Guillaume and Ravetti (17)**. This empirical model uses four quality parameters – induction time, DAGs, FFA Factor (derived from FFA) and PPP – to identify a best-before date (in months) using the lowest value obtained from the following three formulas:

(a) *Hours of induction time at 110°C*

$$(b) \frac{DAGs - 35\%}{FFA \text{ Factor}}$$

FFA factor = 1.7% (if FFA < 0.4%); 2.1% (if FFA > 0.4% and < 0.6%); or 2.5% (if FFA > 0.6%)

$$(c) \frac{17\% - PPPs}{0.6\%}$$

This model recognizes that induction time generally correlates with olive oil fatty acid profiles and antioxidant content. DAGs and PPP have been shown to be predictable and change linearly with time whereas FFA provides a value for the initial oil quality and does not change significantly under proper storage conditions. These four quality parameters represent different factors that can influence olive oil shelf life.

To develop this model, the research team analyzed 118 samples for FFA, PV, UV absorbance, PPP, DAGs and sensory during a 30-month period. The samples were stored in a dark environment at $18^{\circ}\text{C} \pm 2^{\circ}\text{C}$ (61 - 68°F), and tested immediately after reaching their estimated best-before date. Of the 118 samples, only one sample (0.8% of total samples) exceeded the Australian limit of 0.8% for FFA; no sample failed the Australian limit for PV (20 meq O₂/kg) or K₂₃₂ (2.50 K^{1%}_{1cm}); two samples (1.7%) failed K₂₇₀ limit of 0.22 K^{1%}_{1cm}; twelve samples (10.2%) failed the Australian/California limit of 17% for PPP; six samples (5.1%) failed the Australian/California limit of 35% for DAGs; and ten samples (8.5%) failed sensory. In addition to testing 118 samples at the end of shelf life under controlled storage condition, 20 samples with predicted shelf life were randomly collected from different retailers every 3 months during a 30-month period (200 samples in total). Only one sample (0.5% out of 200 samples) exceeded the limit for K₂₇₀ and two samples (1%) exceeded the limit for DAGs at their predicted best before date. By recalculating and comparing the actual best before date and predicted date, the data suggested that producers may want to deduct 1-2 months from the best- before date given from the model to compensate for the potential exposure to heat and light during transportation, storage and display on the retail shelves.

A major advantage of this model is that a California producer can adopt it easily by requesting that a qualified

laboratory conduct induction time analysis for a sample in addition to analysis of FFA, PPP and DAGs as required by California standards. Other advantages of this model are that it included sensory evaluation and was validated on 100+ commercially packaged samples during its development; the calculations are simple and straightforward and yield clear output. Disadvantage is that modification to the predicted time are necessary when storage condition is not ideal, however, this would be true for any models that are designed for the ideal packaging and storage conditions for olive oil shelf life (Table 2).

2. **Aparicio-Ruiz et al (18).** This kinetic prediction model is based on PPP, which is a chlorophyll pigments parameter. Chlorophyll pigments are good aging indicators because they change predictably with time under specific temperatures. The pigment profile is sensitive to small amounts of degradation, which would eventually take place in an extra virgin olive oil even under optimal storage conditions. During storage, pheophytin *a* (PP) degrades to pyropheophytin *a* (PPP), which is a compound that is not observed during olive oil extraction. The ratio of these two compounds therefore is a useful parameter to track olive oil degradation over time.

In developing this model, the research team stored six single-cultivar virgin olive oil samples (cv. Blanqueta, Arbequina, Cornicabra and Picual) in 65 mL amber glass jars with 3% (v/v) headspace, in the dark at room temperature. The monthly temperatures range from 10.4°C (51°F) to 28.6°C (83°F), with the average annual temperature of 19.3 ± 1.9°C (63 - 70°F). The samples were analyzed for chlorophyll pigments every month for one year. Using multivariate statistical analysis, it was found that time, temperature and initial PPP were the main variables that affected PPP prediction for shelf life. A mathematical model to predicted PPP as a function of time and temperature was developed as shown below:

$$\%[\text{PPP}] (t) = \frac{\frac{e^{(\alpha_1 - \beta_1/T)}[\text{PP}]_0}{e^{(\alpha_2 - \beta_2/T)} - e^{(\alpha_1 - \beta_1/T)}} \left[e^{-(e^{(\alpha_1 - \beta_1/T)})t} - e^{-(e^{(\alpha_2 - \beta_2/T)})t} \right]}{[\text{PP}]_0 e^{-(e^{(\alpha_1 - \beta_1/T)})t} + \frac{e^{(\alpha_1 - \beta_1/T)}[\text{PP}]_0}{e^{(\alpha_2 - \beta_2/T)} - e^{(\alpha_1 - \beta_1/T)}} \left[e^{-(e^{(\alpha_1 - \beta_1/T)})t} - e^{-(e^{(\alpha_2 - \beta_2/T)})t} \right]}$$

In this formula, [PP]₀ is the initial concentration of PP while [PPP] is the concentration of PPP over time, other values such as α₁, β₁, α₂ and β₂ are related to kinetic constants and are protected by industrial license according to the authors. PPP at any time point can be calculated if the initial PPP and storage temperature are known.

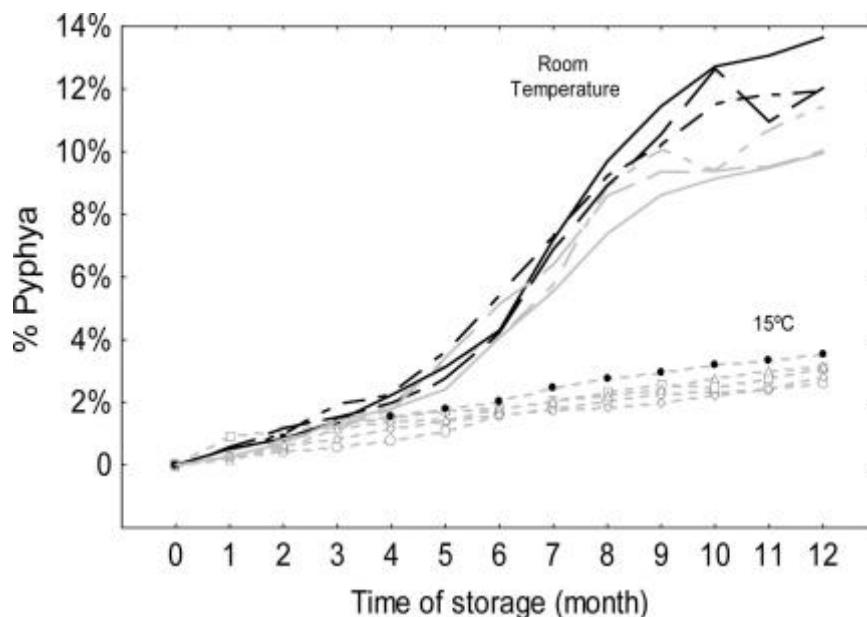


Figure 2. Change of PPP in six monovarieties of VOO (shown in dotted and solid lines) stored at 15°C (59°F) and at room temperature (RT) during a year (18).

Figure 2 shows the change of PPP under a well-controlled storage temperature of 15°C (59°F) and room temperature (RT) for six monovarieties of VOO samples studied. Overall, PPP increased under both temperatures, indicating the degradation of olive oil quality occurred over time. However, it is clear that the same samples stored at RT obtained a significant increase in PPP, especially during summer time (6-8 storage months) when room temperature was higher. The development of this parameter tended to be linear with a smaller slope throughout the entire storage period at 15°C (59°F). This finding confirms the important impact of temperature on PPP generation over time which should be taken into consideration when developing the kinetic model.

Figure 3 shows the predicted PPP for VOO stored at different temperatures (15°C - 35°C/59 - 95°F). The authors suggested that the PPP acceptable limit could be set at 14% (line in red) which would allow oils to have one year of shelf life if stored under 22°C (72°F). However, this value seems arbitrary as the paper did not take into account other chemical parameters or sensory results.

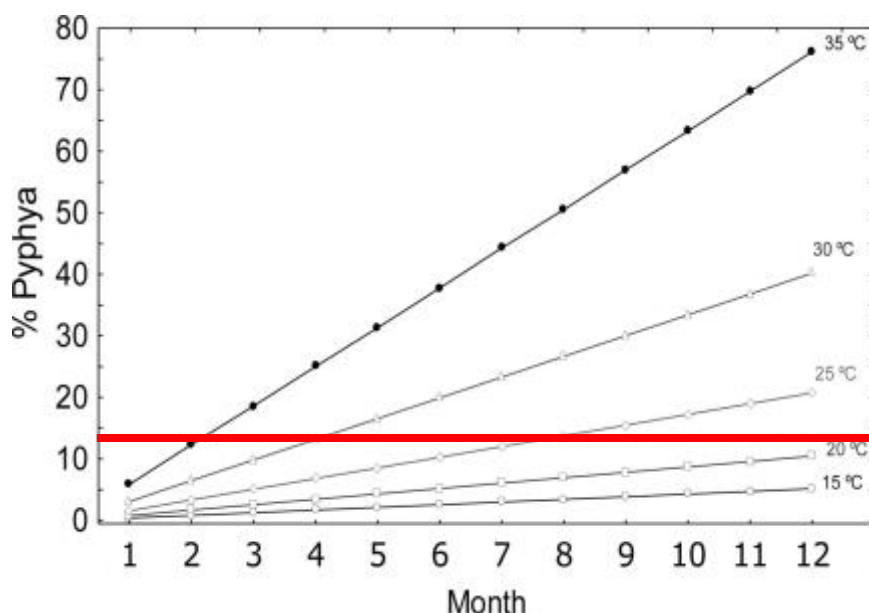


Figure 3. Predicted PPP during one year of storage at temperatures between 15°C and 35°C (59°F and 95°F) (18).

In a follow-up study published in 2014 (19), the same research team applied their prediction model to single-cultivar olive oils (cv. Arbequina) with various levels of initial PPP at bottling. The samples were stored at different average annual temperatures, ranging from 10°C (50°F) to 16°C (61°F). The authors concluded that the initial value of PPP is an important variable to be included when using this model. Table 3 shows shelf life (in months) for olive oils stored at 10°C (50°F) and 16°C (61°F) before reaching the Australian/California limit for PPP of 17%. For example, if PPP is 0.64% at bottling, the oil will have more than 36 months and 21 months before it reaches the limit of 17% if it is stored at 10°C (50°F) and 16°C (61°F), respectively. These temperatures are likely to be cooler than the actual storage temperature, thus a follow up study with oil stored at a typical store shelf temperature is recommended.

Table 3. Shelf life (in months) for oils stored at 10°C (50°F) and 16°C (61°F) before reaching Australian/California limit of 17%.

PPP AT BOTTLING	STORED AT 10°C (50°F)	STORED AT 16°C (61°F)
0.64	>36	21
1.35	>36	20
3.26	>36	19
7.06	34	16
8.66	30	10

The advantages of this model are that it only consists of two chlorophyll pigments and can be used to track the changes of storage temperature and to detect undesired storage condition based on the rate of pyropheophytinization. On the other hand, the model can be benefited from including other quality parameters of the initial samples during its development. Another disadvantage of this model is the complicated calculations which include kinetic constants that are not readily available and the actual quantification of PP and PPP which is not required in California standards.

3. **Psomiadou et al (20)**. This empirical prediction model is based on four parameters (PV, α -tocopherol, total phenols and total chlorophylls) to yield an oxidative stability index (OSI) developed from this formula:

$$OSI = 5.081 + 0.0102(\alpha\text{-tocopherol}) - 0.364(PV) + 0.0477(\text{total chlorophyll}) + 0.0259(\text{total phenols})$$

To establish this model, the research team analyzed 52 Greek VOO samples (cv. Koroneiki) for FFA, PV, UV absorbance, fatty acid profile (for the ratio of unsaturated and saturated fatty acids), α -tocopherol, total phenols and total chlorophylls. Through statistical analysis, the research team selected PV, α -tocopherol, total phenols and total chlorophylls to be the most important factors that affected OSI and included in the model.

The advantages of this model are that the effect of many oxidative parameters on oils from different crop years was examined, following by validation on another 13 samples, and uses a simple calculation. However, while this model gives useful information regarding the oil stability which impacts shelf life directly, it would require producers to incur the expense for three tests (α -tocopherol, total phenols and total chlorophylls) that are not currently required in California standards. Producers can request OSI analysis (by Rancimat) for less of the cost than each of these three tests. Other disadvantages of this model include that the correlation between OSI and shelf life was unclear and sensory evaluation was not included during its development.

4. **Pagliarini et al. (5)**. This empirical/kinetic model uses induction time, hydroxytyrosol, and tyrosol to predict the time (in days) to reach an acceptable limit of 2.1 for K_{232} .

The research team analyzed a total of 37 samples from five different lots which are categorized in Table 4. The samples were subjected to different bottling, transport and storage conditions in supermarkets, although the authors found that the stability of the oil was not significantly affected. This could be due to reasons that 1) the oil was stored properly in the tanks at processing facility in Italy (OL.MA.) before getting bottled; 2) the oil did not experience extreme travel stress during transportation to either Italian supermarket or Australia supermarket; 3) while the oil was stored in supermarkets, the uncontrolled light and temperature were still in favor of maintaining the quality of olive oil.

Table 4. Lot information in Italian Model.

Lot #	Lot A (reference lot)	Lot B ₁	Lot B ₂	Lot C ₁	Lot C ₂
Time taken from freshly made batch	Immediately after processing	After 77 days of storage in tanks	After 188 days of storage in tanks	After 98 days of storage in tanks	After 188 days of storage in tanks
Bottling	100 mL dark glass, closed w/ screw caps	500 mL dark glass, closed w/ screw caps	500 mL dark glass, closed w/ screw caps	500 mL dark glass, closed w/ screw caps	500 mL dark glass, closed w/ screw caps
Shipping destination after bottling	Processing facility in Italy (OL.MA.)	A supermarket in Australia	A supermarket in Australia	A supermarket in Italy (close to OL.MA.)	A supermarket in Italy (close to OL.MA.)
Storage condition at destination	In the dark at 20°C (68°F)	Uncontrolled light and temperature	Uncontrolled light and temperature	Uncontrolled light and temperature	Uncontrolled light and temperature
Storage period at destination	21 months	16 months	14 months	17 months	14 months

The research team tracked the changes in oil during storage with 21 physiochemical parameters and sensory analysis and via multivariate analysis procedure, it was concluded that the most significant parameters were

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K_{232} , induction time, chlorophyll, carotenoid, α -tocopherol, hydroxytyrosol, and tyrosol. Since the only parameter that had established limit in the standards was K_{232} , three empirical models were set up to predict the time to reach a given value for K_{232} and 2.1 was chosen as a reference value:

$$t = 1130.84 \ln(\text{induction time}) - 2388.13$$

$$t = 329.02 - 38.11(\text{hydroxytyrosol})$$

$$t = 580.34 - 68.11(\text{tyrosol})$$

In these formulas t is the time (in days) to reach an acceptable limit of 2.1 for K_{232} . According to the authors, this model underestimates the experimental storage time by 20 days for Rancimat induction time, 10 days for hydroxytyrosol content and 5 days for tyrosol content.

The advantages of this model are that sensory evaluations were taken into account during model development, as well as effect of time bottling, travel stress, and storage conditions. While these three models consist of simple calculations, the output of estimated time is when K_{232} reaches 2.1 instead of 2.4 as stated so the results may not be reliable in its current form. The stability of EVOO depends on many variables including the initial quality, cultivar, ripening degree, processing/storage conditions which were not taken into full account. In addition, the model would require California producers to incur the expense for tests that are not included in California standards.

Four shelf life prediction models are further summarized in Table 5 with information on required tests/parameters (input), estimated shelf life/storage time (output), pros and cons.

Table 5. Summary of shelf life prediction models.

	Input	Output	Pros	Cons
Guillaume and Ravetti	Induction time FFA PPP DAGs	Shelf life in months using the lowest value obtained from the three formulas	<ul style="list-style-type: none"> • Simple calculation. ▪ Uses quality parameters required in California standards. ▪ Sensory evaluation was taken into account during model development. ▪ Validated on 100+ commercially packaged samples. 	<ul style="list-style-type: none"> ▪ Requires modification when storage condition is not ideal.
Aparicio-Ruiz et al.	Initial PPP storage temperature Time	Predicted PPP at given time	<ul style="list-style-type: none"> ▪ Uses only two chlorophyll pigments (PP and PPP). ▪ Provides a tool to track the change of storage temperature and to detect undesired storage condition based on the speed of pyropheophytinization. 	<ul style="list-style-type: none"> ▪ Complicated calculation. ▪ Difficult to obtain kinetic constants used in the equation. ▪ Did not provide information on samples' initial quality other than PPP. ▪ Did not provide correlation with sensory evaluation data. ▪ Needs to quantify actual concentration of PP and PPP which is not required in California standards.
Psomiadou et al.	α -Tocopherol PV Total chlorophylls Total phenols	Oil Stability Index (OSI)	<ul style="list-style-type: none"> ▪ Simple calculation. ▪ The effect of various components on the oxidative stability was examined. ▪ Tests conducted on 52 samples from different crop years and the model was validated on another 13 samples. 	<ul style="list-style-type: none"> ▪ Three out of four needed tests are not required in California standards. ▪ The correlation between OSI and shelf life was not clear. ▪ Did not provide correlation with sensory evaluation data.
Pagliarini et al.	Induction time Hydroxytyrosol Tyrosol	Time in days before the oil reaches $K_{232} = 2.1$	<ul style="list-style-type: none"> ▪ Simple calculation. ▪ Sensory evaluation was taken into account during model development. ▪ Effect of bottling time, travel stress, and storage conditions were taken into consideration. 	<ul style="list-style-type: none"> ▪ Tests are not required in California standards. ▪ Did not provide quality information on samples from lots B and C before bottling. ▪ Estimated storage time is when $K_{232} = 2.1$ instead of 2.4 (upper limit of K_{232} for EVOO in California standards).

CONCLUSIONS

EVOO quality can be safeguarded by having proper packaging, storing in the dark and cool place and marking with an accurate best before date. Currently in literature, common parameters that are being used to track the changes in olive oil include free fatty acidity (FFA), peroxide value (PV), ultraviolet absorbance (UV), 1, 2-diacylglycerols (DAGs), pyropheophytins (PPP), sensory, induction time, total phenols and volatiles. A mathematical model for tracking deterioration using sensitive and accurate quality parameters is a powerful and affordable tool for accurately predicting olive oil shelf life.

However, the literature provides limited practical information that can be adopted by olive oil industry. We summarized four models have the most potential to be implemented by the California producers; the model developed by Guillaume and Ravetti has the most advantages with the fewest disadvantages and it could be the most easily adopted. However, each of the model can be benefited from further study with rigorous experimental design and wide range of samples. To establish a rigorous and systematic model for shelf life assessment, the most urgent tasks are to remove unnecessary parameters and to confirm the acceptable limits without losing the predictive ability and accuracy. By reducing parameters used in the model, shelf life assessment processing time and cost are also reduced. Since sensory evaluation remains to be one of the most sensitive methods for olive oil quality and freshness, a working model should be calibrated with sensory analysis and could complement sensory analysis for olive oil freshness evaluation in the future.

In addition, it is important to continue developing and fine-tuning accelerated methods to minimize their tendency for over-prediction or under-prediction of actual shelf life. Temperature, airflow rate and oil sample size have a significant effect on shelf life prediction. More experiments are necessary to optimize the operational parameters in the Rancimat method to minimize the discrepancy between the real-time shelf life and accelerated prediction.

RECOMMENDATIONS

The OCCC may wish to consider a real-time shelf life study using the Guillaume/Ravetti model and/or model to determine its utility and necessary modification for California oils. Accelerated methods such as the usage of Rancimat need to be optimized to minimize the discrepancy between the real-time shelf life and accelerated prediction for predicting shelf life of olive oils.

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