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Shelf-life predictions for packaged olive oil based on simulations

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Abstract

A descriptive model was applied to study the shelf life of packaged olive oil, based on 24 month simulations and for various combinations of storage conditions close to the "real-life" situations. The major factors influencing oxidation (namely light, oxygen permeability of packaging materials and temperature of storage), were combined in various case studies otherwise requiring time-consuming experiments. The time evolution of the possibility for packaged olive oil not to reach the end of its shelf life and the month, after bottling, at which this possibility becomes 70%, 50% and 30%, were presented. It was found that exposure of packaged olive oil to light in continuous or alternating patterns should be avoided since, even for a short time, it could significantly stimulate the oxidative degradation caused only by elevated temperatures and presence of oxygen. In general, all the packaging materials tested could be considered adequate for preserving the quality of olive oil under a variety of storage conditions. Plastic containers had a particularly stronger protective role when oil was stored light, while glass was the most protective material when oil was stored in the dark.

Keywords: Oxidation; Olive oil; Packaging; Storage conditions; Shelf-life prediction; Modeling

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1. Introduction

Packaging of olive oil may allow its worldwide distribution and retention of quality for an extended period of time. The type of material (plastics, glass, tin) the storage conditions (light, temperature) and the storage period can significantly influence the quality of olive oil. Changes have been reported in relation to acidity and carbonyl compounds produced, reduction of the α-tocopherol content, volatile compounds evolved and sensory changes of the oil (Gutierrez, Herrera, & Gutierez, 1988; Olias & Gutiérrez, 1971; Tawfik & Huyghebaert, 1999). Although significant results have been obtained for the factors affecting quality, a further investigation by choosing different conditions and combinations of storage, and an evaluation of the ability of new polymers to extend the shelf life and overall quality of olive oil, has been a necessity (Tawfik & Huyghebaert, 1999).

After microbial spoilage, oxidation, leading to overt rancidity, is the second most important cause of food spoilage (Lindley, 1998). Although free radical triplet oxygen is the primary mechanism for the formation of volatile flavour compounds in edible oils, oxidation due to photosensitized singlet oxygen, initiated by chlorophyll, has a significant role in the initiation of lipid oxidation (Nawar, 1996).

Besides the comprehensive experimental work on oxidation of olive oil, only a limited number of related mathematical models have been presented in the literature. Based on the reaction kinetics of the food and the active ingredients, the permeability of the film, and the mass transfer rate within the product in the presence or absence of oxygen absorbers, Dekker, Kramer, van Beest, and Luning (2002) presented a model to predict

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Nomenclature

Latin letters/symbols

C_i	concentration of species i
$\langle C_i \rangle^{\text{oil}}$	spatially averaged concentration of species i in the oil-phase
$C_i^{\infty,\text{in}}$	initial concentration of species i at the inner surface of the packaging material
$D_{i,\text{wall}}$, $D_{i,\text{mix}}$	diffusion coefficients of species i inside the packaging material and the olive oil-phase, respectively
k_a, k_b, k_c	constants for reaction rates (2a), (2b) and (3), respectively
k_{H}	Henry's constant
p_{O_2}	oxygen partial pressure
P_{wall}	permeability of the packaging material
$P_{\text{safe}}(t)$	possibility for the stored olive oil after a time period t to reach the end of the shelf-life
t	time
x	spatial coordinate

Greek letters/symbols

ξ	light indicator ($\xi = 0$ corresponds to dark, $\xi = 1$ corresponds to light)

the development of hydroperoxides as a function of both time and location in the package. They proposed the use of their model as an initial step before performing actual shelf-life experiments, as a way to achieve a quick estimation of the product's response. A generalized approach to describe oxidation was presented by Del Nobile, Ambrosino, Sacchi, and Masi (2002) and Del Nobile, Bove et al. (2003) for olive oil packaged in bottles of various shapes and materials. Their parametric analysis was limited in the dimensions of the bottles, as well as in the approximation of the packaging materials oxygen permeability, without any further refinement in terms of storage conditions, namely temperature and light.

Based on the experimentally obtained data during a shelf-life estimation study for olive oil packaged in different packaging materials and stored under different combinations of temperatures and availability of light, Kanavouras, Hernandez-Münoz, Coutelieris, and Selke (2004) calculated the growth rate of various oxidations deriving off-flavour compounds, as well as the equilibrium oxidation reaction constants. Their model, based on the experimental work presented, was limited to chemical processes occurring inside the oil mass with the inadequacy of not incorporating the mass transport of the most oxidation-characteristic compounds due to diffusion. In a consequent study, a predictive mathematical model was introduced to describe the mass transport from and to the oil phase through various packaging materials for several temperatures and light availability storage conditions (Coutelieris & Kanavouras, 2005). Based on the validated simulations and by introducing the possibility of the packaged olive oil not reaching the end of its shelf life after a certain period of time, the researchers drew conclusions on the qualitative changes of packaged olive oil stored under various conditions for prolonged periods of time.

It is, in general, recognized as a time- and effort-consuming approach to apply lab-scale experiments in order to study the effects of more complicated storage environments, which, nonetheless, are more likely conditions during the distribution cycle. Therefore, the present study applies the model, developed previously by Coutelieris and Kanavouras (2005), to extra virgin olive oil, packaged in various packaging materials and stored under a wide range of storage conditions, in order to achieve on a more flexible and comprehensive shelf-life evaluation.

2. Materials and methods

The *theoretical background* of this work was based on the assumption that convection is a non-existing phenomenon in the quiescent oil, and therefore the transport of oxygen entering the bottle can be described by the diffusion equation:

$$\frac{\partial C_{\text{O}_2}}{\partial t} = D_{\text{O}_2,\text{wall}} \frac{\partial^2 C_{\text{O}_2}}{\partial x^2}, \tag{1}$$

where the oxygen diffusion coefficient through the packaging material, $D_{\text{O}_2,\text{wall}} = k_{\text{H}} p_{\text{O}_2} P_{\text{wall}}$, is given by Henry's law (Crank, 1975).

The chemical reactions inside the bottle can be summarized as follows:

$$^3\text{O}_2 \xrightarrow[hv]{k_\text{a}} {}^1\text{O}_2 \tag{2a}$$

$$\text{RH} + {}^1\text{O}_2 \xrightarrow{k_\text{b}} \text{ROOH} \tag{2b}$$

$$\text{RH} + \text{O}_2 \xrightarrow{k_\text{c}} \text{ROOH} \tag{3}$$

with RH being any fatty acid serving as the oxidation substrate, ROOH the derived hydroperoxide, and k_a, k_b and k_c the reaction constants influenced only by temperature (Kanavouras et al., 2004). The simultaneously occurring reactions (2a) and (2b) take place only in the presence of light. Hydroperoxides are eventually transformed to off-flavour compounds, among which the most prominent one with the greatest impact on the sensory evaluation of the olive oil is hexanal. We can quite justifiably assume that C_{ROOH} is actually C_{hexanal}, which is, in the end, the one most likely to be sorbed by the polymeric packaging materials ("scalping"). Therefore, hexanal will from now on be considered as the major final oxidation process product that may be present at various concentrations in the oil. By assuming quasi-steady state for the intermediate product $^1O_2$ (Atkins & de Paula, 2002), the following set of differential equations can be written to express the mass transport through the oil-phase:

$$\frac{\partial C_{O_2}}{\partial t} = D_{O_2,\mathrm{mix}} \frac{\partial^2 C_{O_2}}{\partial x^2} - \xi k_a C_{O_2} - k_c C_{O_2}\left(C_{O_2}^{\infty,\mathrm{in}} - C_{O_2} - C_{\mathrm{hexanal}}\right), \tag{4}$$

$$\frac{\partial C_{\mathrm{hexanal}}}{\partial t} = D_{\mathrm{hexanal,mix}} \frac{\partial^2 C_{\mathrm{hexanal}}}{\partial x^2} + \xi k_a C_{O_2} + k_c C_{O_2}\left(C_{O_2}^{\infty,\mathrm{in}} - C_{O_2} - C_{\mathrm{hexanal}}\right). \tag{5}$$

The diffusion of the adsorbed hexanal trough the packaging material was according to the following equation:

$$\frac{\partial C_{\mathrm{hexanal}}}{\partial t} = D_{\mathrm{hexanal,wall}} \frac{\partial^2 C_{\mathrm{hexanal}}}{\partial x^2}. \tag{6}$$

The above system has to be integrated by assuming a constant initial spatial profile of the concentrations, axial symmetry, continuity of the oxygen concentration, as well as typical Langmuir-type adsorption of hexanal by the packaging material (Coutelieris, Kainourgiakis, & Stubos, 2003). A detailed mathematical description of the above-mentioned initial and boundary conditions is given elsewhere (Coutelieris & Kanavouras, 2005).

In order to estimate the time needed for the packaged olive oil to reach a quality threshold, we need initially to introduce a certain value of C_{hexanal} as an upper limit for the quality acceptance. Accordingly, the probability of the olive oil reaching the end of its shelf life during a certain time period, is analogous to the area above the arbitrarily defined quality threshold and below the concentration curve. Since the above-mentioned surfaces can be expressed by integrals, we can define the probability, P_{safe}, for the oil not to reach the end of its shelf life during the time period $[t_1, t_2]$ as:

$$P_{\mathrm{safe}} = 1 - \frac{\int_{t_1}^{t_2} \langle C_{\mathrm{hexanal}} \rangle (t)\, \mathrm{d}t}{\int_{0}^{t_2} \langle C_{\mathrm{hexanal}} \rangle (t)\, \mathrm{d}t}, \tag{7}$$

where the brackets denote spatial averaging, t_1 is the time when C_{hexanal} reaches the critical value and the upper edge of the integrals, t_2, could be any time period. In this study, $t_2 = 24$ months. In general, P_{safe} is a simply estimated quality indicator, dependent on the evolution history of the compound in question through a single value that allows an extensive analysis of experimental data and easy-to-make comparisons. Further on, it will be employed in this study for the analysis of the results.

Portuguese organic extra virgin olive oil was placed in 500 ml PET, 500 ml PVC (Novapack, Co., Paris, IL, USA) or 500 ml glass bottles (Fisher Scientific Co., NJ, USA). *The properties of the packaging materials* were previously evaluated (Kanavouras et al., 2004). For each packaging material, 144 bottles were evaluated. Half of the bottles were stored in the dark and the other half were exposed to fluorescent light (four 40 W fluorescent light bulbs were placed at 30 cm above the bottles), all in controlled environment chambers at 15, 30 or 40 °C. For every treatment, two bottles were removed in each sampling day and olive oil from each bottle was analysed in triplicate. *Separation and identification of hexanal* were according to the previously developed methodology (Kanavouras et al., 2004). *Statistical analysis* was performed using commercial software (SAS® Proprietary Software Release 8.2, TS2M0, SAS Institute Inc., Cary, NC, USA) to determine differences between treatments for the rate of evolution of hexanal. GLM analysis was applied and the Tukey and Duncan tests were implemented for separating the means of GC area changes among the amounts of hexanal at $\alpha = 0.05$.

Regarding the *model*, the boundary value problem previously described by Eqs. (1) and (4)–(6), was localised in space and time using a non-uniform finite-difference scheme with an upwinding numerical algorithm, that involves a typical Newton method for non-linear systems in conjunction with the finite differences scheme. The values for the parameters were taken from the relative literature (Del Nobile, Bove, La Notte, & Sacchi, 2003; Feigenbaum et al., 1991; Hernandez-Munoz, Catala, & Gavara, 1999; Kanavouras et al., 2004; Schumpe & Luhring, 1990; Toi, 1973). When necessary, numerical interpolation (or extrapolation) was applied to the experimentally measured values. The adequacy of the proposed model to safely predict qualitative changes of packaged olive oil has been previously validated (Coutelieris & Kanavouras, 2005).

3. Results and discussion

Characteristic results on the shelf life of the packaged olive oil derived for the selected simulations are presented in Table 1. Combinations of the storage factors were chosen in order to investigate the predictive ability of the model for "real-life" storage conditions, otherwise hardly attainable by experimental procedures. According to this concept, an overall simulation period of 24 months for the following cases was considered:

Table 1
Summary of the various combined storage conditions against which the model was tested

Cases	Exposure to light	Temperature (°C)
1A	Dark	15(4M)–30(4M)–40(4M)–15(4M)–30(4M)–40(4M)
1B	Light	15(4M)–30(4M)–40(4M)–15(4M)–30(4M)–40(4M)
2A	12-h Dark/12-h light	15
2B	12-h Dark/12-h light	30
2C	12-h Dark/12-h light	40
3A	1M(dark) + 23M(12-h dark/12-h light)	15
3B	2M(dark) + 22M(12-h dark/12-h light)	15
3C	3M(dark) + 21M(12-h dark/12-h light)	15
4	1M(dark) + 23M(12-h dark/12-h light)	15(4M)–30(4M)–40(4M)–15(4M)–30(4M)–40(4M)

M, months; h, hours (see text for further explanation).

- Case 1A: oil was stored at temperatures of 15, 30 and 40 °C, alternating every 4 months, and under continuous dark.
- Case 1B: the same temperature pattern as in case 1A, but with continuous light exposure.
- Case 2A: every 12 h daily alteration of light and dark, at 15 °C for 24 months.
- Case 2B: every 12 h daily alteration of light and dark, at 30 °C for 24 months.
- Case 2C: every 12 h daily alteration of light and dark, at 40 °C for 24 months.
- Case 3A: the same as in case 2A, but with a 1-month initial period of dark.
- Case 3B: the same as in case 3A, but the initial period of dark was 2 months.
- Case 3C: the same as in case 3A, but the initial period of dark was 3 months.
- Case 4: case 3A, modified by a pattern of alternating temperatures (15, 30, 40 °C), every 4 months.

The presented cases were chosen in order to accumulate the influence of the most oxidation-influencing parameters, such as light (case 1A and B), temperature (case 2A–C) and initial period that the oil was stored in the dark (case 3A–C). Case 4 demonstrates an example of characteristic storage conditions combined in a more realistic way. Comparisons within cases can be more clearly seen in Table 2, where the months after which the oil has 30%, 50% and 70% possibility of not reaching its shelf-life (P_{safe}) are presented.

Fig. 1 shows P_{safe} as a function of time, for the cases 1A and 1B. The abundant effect of light on the quality of packaged olive oil can be clearly concluded through an apparently logarithmic decay of the P_{safe} during the initial 6 months of storage (Fig. 1(b)). This trend may denote the corresponding logarithmic increase in the oxidation-deriving by-products, most likely due to the similar hydroperoxide production patterns, stimulated by the helpful abundance of light (Angelo, 1996; Labuza, 1971). The influence of the plastic packaging materials on the quality of olive oil stored under continuous light did not seem to significantly differ among PET and PVC. Although both plastic packaging materials adequately prevent quality losses in the oil ($P_{\text{safe}} > 67\%$ for the examined period of 24 months), PVC presented a slightly better protection ability than PET, most likely due to its difference in oxygen transmission properties. Glass, on the other hand, showed a rather clear protective role in dark storage conditions, since it is impermeable to oxygen. The above presented data are in accordance with previously reported studies on the oxidation of olive oil stored under light and dark conditions in various packaging materials (Gutierrez, 1975; Gutierrez et al., 1988; Kiritsakis & Dugan, 1985; Min, 1998; Kanavouras et al., 2004).

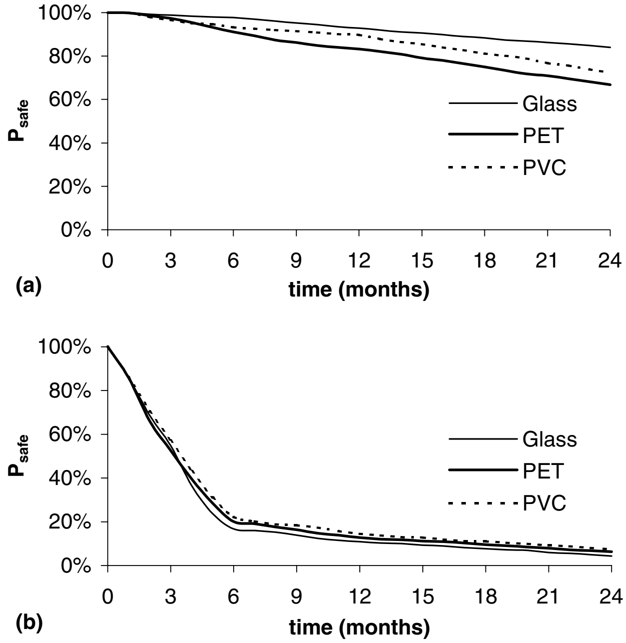

Fig. 1. Time evolution of P_{safe} for cases 1A and 1B.

The influence of temperature on the retention of the packaged olive oil quality is shown in Fig. 2, where the P_{safe} values for the packaged olive oil stored under alternating light and dark conditions, and at a constant temperature of 15, 30 or 40 °C, are given as a function of time (see also Table 2). In this group of cases the light exposure was chosen to be the same as in case 1, in order

Table 2
The critical month at which P_{safe} reached 30%, 50% or 70%

	Case 1A	Case 1B	Case 2A	Case 2B	Case 2C	Case 3A	Case 3B	Case 3C	Case 4
70%									
Glass	>24	1–2	2–3	2–3	1–2	4–5	7	8–9	2–3
PET	21–22	1–2	11	8	3–4	13–14	16	17–18	5
PVC	>24	2	15–16	11–12	5	16–17	19–20	22	7–8
50%									
Glass	>24	3–4	8	6–7	3–4	10–11	12–13	14–15	5
PET	>24	3–4	>24	17–18	10–11	>24	>24	>24	13–14
PVC	>24	3–4	>24	22–23	13–14	>24	>24	>24	16
30%									
Glass	>24	4–5	21	17	11	22–23	>24	>24	13
PET	>24	4–5	>24	>24	24	>24	>24	>24	>24
PVC	>24	5–6	>24	>24	>24	>24	>24	>24	>24

to isolate the effect of temperature on the P_{safe}. The combined effect of elevated temperature (40 °C) and the presence of light revealed an initial highly stimulated oxidation for oil stored in all packaging materials. Results in Fig. 2 are similar to those presented in Fig. 1(b), although at a much lower amplitude. For the same storage temperature, glass was a significantly less protective material for packaged olive oil under the presented light conditions. PVC showed a greater protective role, although not that different from PET, most probably due to its higher oxygen diffusivity. The alternating presence of light had clearly reduced the possibility of the oil reaching the end of its shelf-life, compared to continuous light exposure (see Fig. 1(b)). In Fig. 3, the critical time period at which the olive oil reached the end of its self life for $P_{safe} = 70\%$, is presented for a quantitative description of the quality. In this study, the critical time period was defined by the assumption that $P_{safe} = 70\%$ which consequently does not correspond to a real end-point in the quality reduction. In that sense, using the activation energy, to describe the rate of the quality decrement in packaged olive oil, could not be accurate and, therefore, the use of an Arrhenius-type model for the data in Table 2, is not recommended. It can be concluded that oil packaged in glass seems to be less "sensitive" to temperature variation. Regarding the slopes of the lines for PET and PVC in Fig. 3, a similar classification of their protective roles can be concluded for these materials as well (see Fig. 2 for comparison).

For the cases 3A–C, the P_{safe} values are presented in Fig. 4 as a function of time. In accordance with Fig. 1(a), where storage in the dark corresponded to lower concentrations for hexanal in the oil-phase, longer initial dark periods, in case 3, corresponded to higher P_{safe}

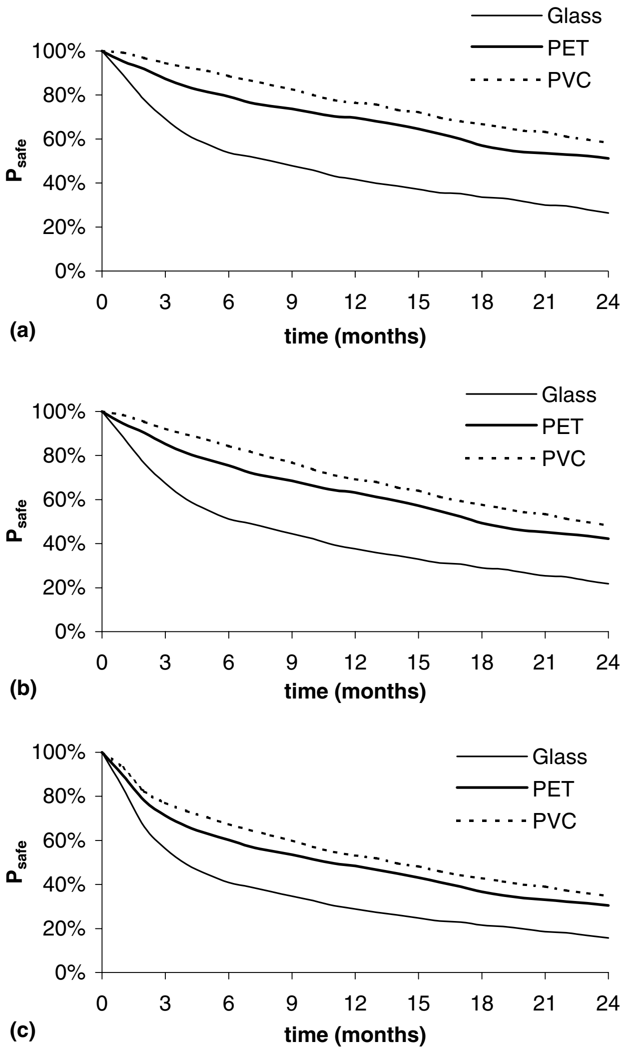

Fig. 2. Time evolution of P_{safe} for cases 2A, 2B and 2C.

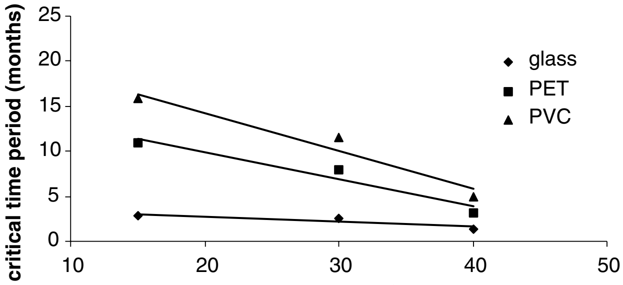

Fig. 3. Effect of temperature variance on the shelf life for the cases 2A–C.

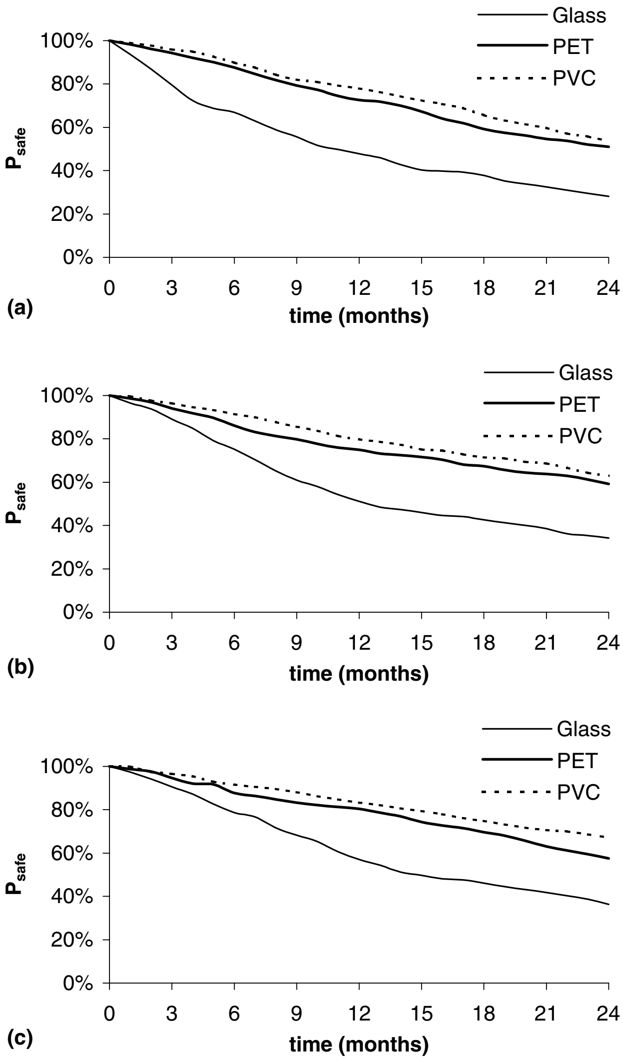

Fig. 4. Time evolution of P_{safe} for cases 3A, 3B and 3C.

values. In addition, the initial delay in the reduction of the P_{safe} was analogous to the time the product was continuously stored in the dark. Packaging materials showed a similar effect on the P_{safe}, independently of the initial dark period. Their protective behaviour followed the previously discussed order.

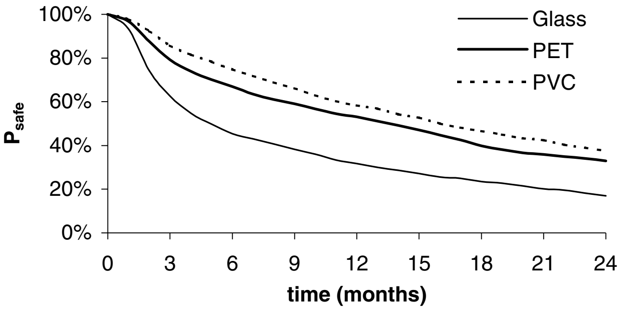

Fig. 5. Time evolution of P_{safe} for case 4.

Case 3A can be concluded to be the most aggressive storage conditions for packaged olive oil, based on the lower P_{safe} among the cases 3A–C. These conditions were further modified to include (at every 4 months) a temperature alternating period for each selected temperature, denoted as case 4 in Table 1. Fig. 5 shows clearer overall combined effect of temperature, light and packaging materials. During the storage of packaged olive oil, elevated temperatures clearly affected the shelf life compared to continuous exposure at 15 °C (case 3A). The initial delay in the decrement of P_{safe} was similar to case 3. Packaging materials showed similar protective roles as in cases 1 and 2. The synergistic effect of light could easily be seen by comparing Figs. 1(a) and 5, where the presence of light appeared to be more important than the exposure to high temperatures, indicating that photo-oxidation, as an additional source of oxidation by-products, could play an auto-catalytic role in the overall deterioration of the oil.

As previously discussed (Fig. 1(a)), glass had the best performance when oil was stored continuously in the dark while in the presence of light, its protective role was clearly diminished, falling below those of the two plastic materials. Although there was a clear indication of the superiority of glass due to its oxygen barrier properties, plastic materials could apparently provide a better light transmission resistance, and greater protection in the presence of light. That was shown to be of paramount importance for the preservation of olive oil. Even though PVC had better oxygen barrier properties than PET, under all storage conditions, relative differences in the olive oil protection role were not significantly affected by the storage temperatures.

In addition to the results related to the time evolution of the olive oil in terms of P_{safe}, Table 2 shows the critical month during which P_{safe} became 70%, 50% and 30%, respectively, for all the cases. This month indicates the time at which the oil has 30%, 50% or 70% possibility of reaching the end of its shelf life, respectively. The months recorded in this table, for the different cases, followed the same trends as the time evolution results of P_{safe} discussed above.

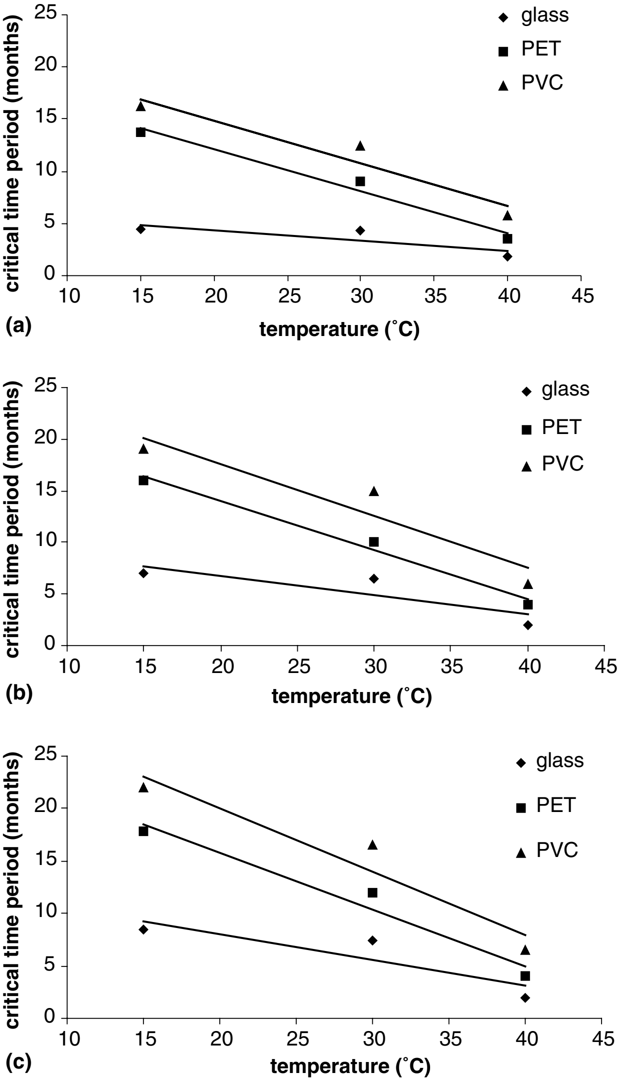

Fig. 6. Effect of temperature variance on the shelf life for different initial storage periods in the dark (6A = 1 month, 6B = 2 months, 6C = 3 months).

To further investigate the influence of temperature variance, in conjunction with the period that the oil was initially stored under dark, Fig. 6 shows the rate of the influence of the combined storage factors on the quality of the oil, i.e., on the time needed for the packaged oil to reach the end of its self life for $P_{\text{safe}} = 70\%$. Data were partially taken from Table 2 and further simulations not included therein. After linear fitting, the slopes of the lines revealed that, the longer the initial storage period in the dark, the higher was the deterioration of the olive oil, as a result of the temperature-enhanced oxidation phenomena, pre-dominating during this period. A further enhanced influence of the temperature variance during the period of exposure under alternating light conditions was also observed. Since the overall effect of light on olive oil appeared to be more significant than temperature, it can be concluded that the temperature variance had a smaller impact on the quality degradation. For a given temperature variance, the exposure to light, following the initial dark period, seemed to make a significant synergistic contribution to the reduction of the shelf-life. Accordingly, it is noteworthy that a prolonged initial storage of packaged olive oil, under similar continuous dark storage conditions, is not a protective treatment equivalent to isothermal storage, especially for low temperature conditions.

4. Conclusions

Reliable estimations of the shelf life of packaged olive oil were obtained for various combinations of storage conditions, close to“real-life” situations, by using a theoretical model supported by experimental results. By using the P_{safe} factor, defined as the possibility of the packaged olive oil not reaching the end of its shelf-life, enough evidence was obtained to support the benefits of storing the olive oil under continuous dark and low temperature conditions. It was found that longer initial storage periods in the dark corresponded to higher P_{safe} values, based on the lower concentrations of hexanal evolved in the oil-phase. Elevated temperatures, although stimulating the deteriorative reactions, were not as significant as light in continuous or alternating patterns. Initial storage of packaged olive oil, in the dark, synergistically interacts with elevated temperature variance toward quality losses, as shown by the corresponding P_{safe} values. All the materials tested could provide sufficient protection to packaged olive oil kept in the dark. In any case, even a short-time exposure of the oil to light should be avoided, since it could significantly stimulate the oxidative degradations, further assisted by elevated temperatures and the presence of oxygen.

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