

Alterations of PET material physical properties during storage of olive oil

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ABSTRACT

Olive oil in PET bottles as well as empty bottles were stored at 15, 30 and 40 °C, at light or dark, for 12 months. Oxygen transmission rate and light transmission were estimated in addition to thermographic parameters, color, crystallinity and sorption. Results indicated that oxygen transmission rates for both the control (empty) and the oil-containing PET bottles depend on time/conditions of storage. Significant differences of thermal properties were observed within the first four months, among PET materials in contact to olive oil and the control ones. Sorption of humidity was also reported for all containers, revealing the effect of the simultaneous presence of olive oil on the results. Apparently, olive oil has a significant impact on the properties of PET, while the reported alterations of its properties may affect the food preservation it self and its prediction methodology if not appropriately considered.

1. Introduction

A number of studies reported the role of packaging materials on the preservation of the olive oil (Cecchi, Passamonti, & Cecchi, 2010; Coutelieres & Kanavouras, 2006; Del Nobile, Ambrosino, Sacchi, & Masi, 2003; Kanavouras, Hernandez-Munoz, & Coutelieres, 2004; Kanavouras & Coutelieres, 2006; Mendez & Falqué, 2007). The oxygen permeability of all polymers is evidently, depended on both temperature and relative humidity (Axelson-Larsson, 1995), different food stimulants (Piergiovanni, Nicoli, Tinelli, & Vestrucci, 1995) and the aroma compounds absorbed by the polymer (Sadler & Braddock, 1990). Del Nobile, Bove, La Notte, and Sacchi (2003), pointed out a significant dependency among the oxygen permeation per unit volume of oil, and the shape and size of the bottle. The results may only refer to those values of $D_{O_2}^{Polym}$ with a higher proximity to the oxygen diffusion coefficient in the PET ($D_{O_2}^{PET}$), while in the case of far more different $D_{O_2}^{Polym}$, the impact of the bottle material on the permeating oxygen qualities, was significantly reduced.

In glassy crystalline polymers, such as polyethylene terephthalate (PET), the isotropic Fickian diffusion of gas molecules is defined by the crystalline regions. The ratio of diffusion is estimated via the different activation energies needed by the diffusing particle to pass through each region (Polyakova, Liu, Schiraldi, Hiltner, & Baer, 2001; Polyakova, Stepanov et al., 2001). Steady-state permeability experiments indicated that the oxygen permeability coefficients for PET films (being $1-6 \times 10^{-19}$ kg m/m² s Pa, at different temperatures and water activities), did not change significantly with water activity, when polymer was exposed at temperatures lower than 23 °C (Auras, Harte, &

Selke, 2004).

Sorption of small quantities of various oil components by the plastic packaging may also alter the plastic's barrier and mechanical properties during the package shelf life (Johansson & Leufvén, 1994; Tock, 1984). Olive oil penetrating the plastic package could possess a plasticizing effect on the polymers, resulting in swelling (Riquet, Wolff, Laoubi, Vergnaud, & Feigenbaum, 1998).

Accordingly, food-packaging inter-relationship and environment-packaging intra relationship, are significant factors of the olive oil's quality. Hence, a need to further investigate and describe the potential alterations of the performance properties of plastic materials, against the capability of the plastic containers to retaining the quality of the olive oil, over a realistic storage period, is still of interest.

This study aims in providing justified evidence for the impact of olive oil and the storage conditions of packed products on the PET properties. The study targets to provide evidently support towards a more precise estimation of the olive oil's preservation. Thus, the objective of this study is to describe changes of the PET polymer, such as thermal properties, oxygen permeation, light transmission and color, when PET bottles were filled with virgin olive oil during one year of storage, as compared to empty ones.

2. Experimental

2.1. Materials

2.1.1. Olive oil

Extra virgin olive oil produced and packed in Portugal, stored in

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green-colored 750 mL glass containers placed in fiberboard boxes, was used for this study.

2.1.2. Polymeric materials

500 mL PET containers previously used for drinking water were used after cleaning and drying with a PP seal-cup, tightly screwed on the bottles' neck.

2.1.3. Filling and storage conditions

Plastic containers were filled with the olive oil under nitrogen gas, to diminish oxygen trapping. No headspace was left while polypropylene caps were used to seal the bottles tight. Two sets of bottles were prepared: one containing the olive oil, and the other one of empty ("control") bottles. According to the experimental design, half of the bottles were covered with Alu-foil and placed in the dark within carton containers, while the rest were placed under fluorescent light provided by 4 × 40 W fluorescent light bulbs, placed 30 cm above the bottles. All bottles were stored within controlled environment chambers at 60% RH, and at 15, 30, or 40 °C. Each month two bottles per treatment were sampled and analyzed in triplicate, over a period of 12 months.

2.1.4. Bottles' preparation

Oil containing bottles were thoroughly cleaned with mild soap and distilled water to remove residual oil, dried in absorbing paper and prepared for analysis.

2.1.5. Oxygen permeability

All bottles were tested for their oxygen permeability using OXTRAN-Twin equipment (MOCON, MN) equipped with a coulometric sensor. Gas mixture consisting of 98% nitrogen and 2% hydrogen was used as a carrier gas. Both oxygen and the carrier gas was flowed at 20 mL/min. Oxygen transmission rate (OTR) measurements were performed at 23 °C. Before entering the testing cell, gases were passing through distilled water so as that at their exit they contained 85% RH. Tests were considered as complete when the oxygen values, recorded every ten minutes, did not differ from previous the measurement by more than 1%. Two bottles were tested per sampling time, in three replicates each. For all permeability calculations a standard and uniform thickness across all samples was considered.

2.1.6. Differential Scanning Calorimetry (DSC)

A differential scanning calorimeter (TA DSC 2920, TA Instruments Inc., New Castle, DE, USA) equipped with an integrator (Universal V2.6B TA) was used for recording the thermal parameters of the polymer. Indium was used to calibrate the equipment. Plastic samples of either oil exposed or control bottles (10–15 µg) were placed within a sealed aluminum disc, treated under a nitrogen flow of 50 mL/min. In order to obtain the melting point (T_m), a heating cycle was applied up to 310 °C at 10 °C/min.

An endothermic base line shift during the initial thermal scanning of the polymers was recorded. Scanning was ended when the temperature reached approximately 20 °C above the temperature of this base line shift. To reveal an actual, reversible glass transition temperature (T_g), a second scanning was performed using the samples from the first scan, following a quick cooling period. The reported T_g of the samples was obtained from that second scanning procedure, while T_m values were taken during the first run.

2.1.7. Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier Transform Infrared Spectroscopy (FTIR) analysis of PET bottles was performed with a spectrometer (Perkin Elmer Spectrum 1000 FT-IR) for wavelength ranges 4400 to 360 cm^{-1} and 7500 to 4500 cm^{-1} .

2.1.8. VIS-light transmittance

Quality and amount of light passing through the oil exposed and

control bottles were evaluated by a spectrophotometer (Perkin Elmer Lambda 25 UV-vis) for the range of 1100–200 nm and analyzed with software (UV-WIN-Lab).

2.1.9. Color

The outer surface of olive oil-exposed and control plastic bottles were tested for color changes during storage. A Hunter Lab Color-Quest Spectrophotometer Colorimeter (Hunter Associates Laboratories Inc., Reston, Virginia, USA), calibrated against green, black and white standards (Standards Gardner Laboratories, Bethesda, Maryland, USA), was used. The L^* , a^* , and b^* chromatic coordinators were recorded using a D65 illuminated at a 2° angle using Color-Quest software (version 2.33, 1987).

2.1.10. Statistical analysis

Commercial software (SAS™ Proprietary Software Release 8.2, TS2M0, SAS Institute Inc., Cary, NC, USA) was used to statistically analyze the data obtained from each analysis to identify the differences among treatments. ANOVA and the Tukey test were implemented for analyzing the mean changes of the parameters at a confidence level of $\alpha = 0.05$.

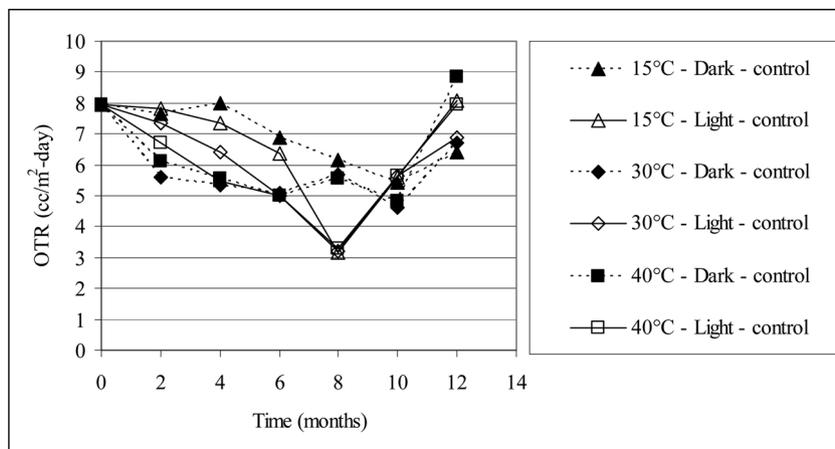
3. Results and discussion

3.1. Oxygen permeability

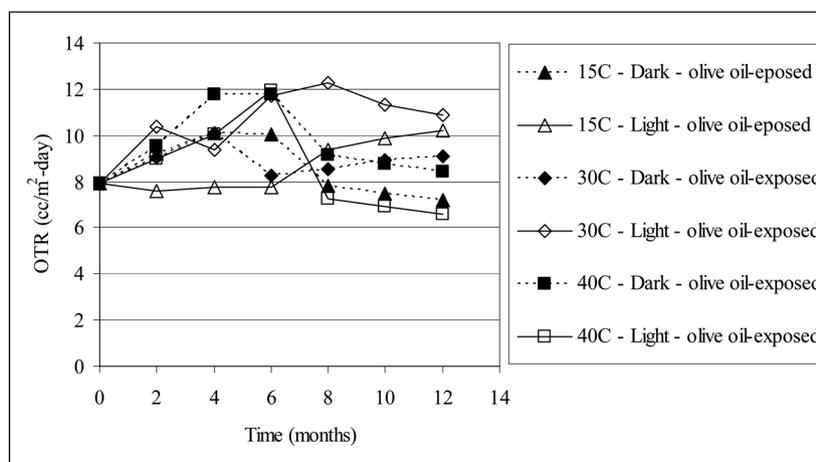
Initial values for oxygen transmission rates (OTR) were 8 $\text{cc}/\text{m}^2 \cdot \text{day}$ for PET materials at 1 Atm. As shown in Fig. 1a & b, the decrease with the storage time of the OTR through PET was more acute at 30 °C and 40 °C than at 15 °C. Oil exposed PET bottles presented higher OTR values than control for the same temperature at a given time point. Differences between OTR values of control and oil-exposed bottles stored at 15 °C were statistically significant for the total storage period studied. For samples stored at 30 °C and 40 °C, significant differences were reported after 12 months of storage. Changes in OTR among control PET bottles stored at 15 °C, 30 °C and 40 °C were statistically significant up to 8 months of storage, but not for the rest 4 months of the storage. Differences in the OTR of oil exposed bottles stored at 15 and 40 °C were significant only after the 8th month of storage. No clear differences could be reported for the oil containing bottles kept at 30 and 40 °C, while bottles stored at 15 °C showed a rather higher OTR after the 6th month, when stored at light. Although the changes for the control bottles were probably related to the effect of the relative humidity in the environmental chambers, the changes in the olive oil-exposed bottles should be attributed to both the presence of humidity and the oil penetrating the polymer.

Results were in agreement with those previously reported by Johansson and Leufvén (1994) that the OTR values increased for amorphous PET films in contact with rapeseed oil, when kept in contact for 40 days, indicating that oil might have acted as a plasticizer dispersing the polymer chains (swelling effect), and increasing the free volume of the polymeric matrix. As a consequence, the diffusion of the oxygen molecules through the polymer increased, resulting in a slight higher OTR for the polymers containing oil compared to the control ones.

The decrease in the OTR for the PET bottles may be explained by the previously reported results of Johansson and Leufvén (1994) who reported that OTR of amorphous PET films was directly depended on the relative humidity. In contrast, for the no oil-contacting films at RH > 55%, in contact to oils showed a higher OTR, possibly due to the reason that water could bind the available polar sites of the polymer and restrict passage of oxygen through it. Furthermore, the free volume of PET polymer was found to grow with exposure of the polymer to increased humidity (from 55% to 100%), (Lightfoot & Xu, 1993; Tatsumi, Ezure, & Ito, 1992). In addition, McGonigle et al. (1999) and McGonigle, Daly, Jenkins, Liggat, and Pethrick (2000), showed that



a



b

Fig. 1. a Oxygen Transmission rate (OTR) for control bottles stored at 15, 30 and 40 °C in dark and light. b) Oxygen Transmission rate (OTR) for oil-exposed PET bottles stored at 15, 30 and 40 °C in dark and light.

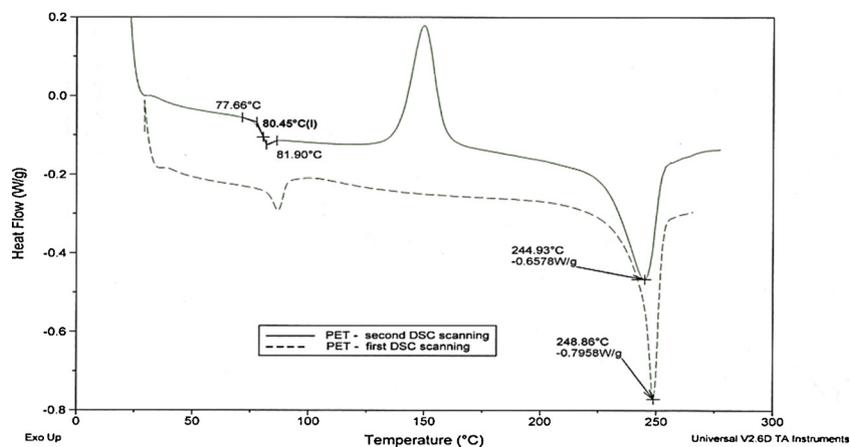


Fig. 2. Differential scanning calorimetry analysis of PET bottles to reveal the real glass transition temperature.

the free volume of PET was not affected during storage at 40 °C and dry conditions (McGonigle et al., 1999; McGonigle et al., 2000 and Hill, Weinhold, Stack, & Tant, 1996). Also, temperature exposure of glassy polymers aged below the T_g , at levels near the T_g or an increase in the polymers' crystalline regions, may influence the tortuosity path of

oxygen through the polymers (Dorkenoo & Peromm, 1999).

Oxygen transport through packaging material is almost exclusively considered a diffusion-driven process, while convection due to pressure difference between the food-contact side of the bottle and the outer environment is rather neglected. On top of the oxygen diffusion

phenomena, this work may constitute an accountable data for calculating the permeability of a plastic container as a function of the material, temperature and light presence, based on pragmatic mass transport processes. Accordingly, when pressure difference ΔP is known, it is rather straightforward to convert the permeability values K to velocity ones u through the well-known Darcy law, as shown below:

$$u = -\frac{K \Delta P}{\mu L_W} \quad (1)$$

where, μ is the viscosity of the oxygen gas and L_W is the thickness of the plastic container. Independent of the material, it was found that velocity is has very low values, even for the most mass-transfer favorable temperature and light conditions, which obviously further indicates that convection is indeed negligible.

3.2. Differential Scanning Calorimetry (DSC)

Typical results of the first and second DSC scanning for PET bottles, can be seen in Fig. 2. Since, the grant majority of the polymers containing polar groups, exhibit a secondary relaxation below their glass transition temperature, often attributed to small-scale local motions such as torsional oscillation of the main chain or rotation of the polar side groups (Bell & Touma, 1996), a second scan was performed. Re-scanning (second DSC scanning) may identify the real, reversible T_g at the 80.45 °C, assuming that no other thermal changes took place prior to the rescanning.

The T_g values for bottles stored at 15 °C were rather close for both scans, while higher initial T_g values were recorded for bottles kept at 30 and 40 °C compared to those of the second scan. Some residual accumulated stresses inside the polymer's mass, most probably formed during the blow molding process, may then relax during storage at various temperatures. Additionally, a relaxation "exothermic" peak has been observed at the range of 100–140 °C, most likely as a consequence of the stretching process during the PET bottle formation (Cakman, White & Spruiell, 1986; McGonigle et al., 1999). Bell and Touma (1996), reported that amorphous polymers in the glassy state have the tendency to undergo a secondary molecular ordering which appears as an irreversible endothermic relaxation peak (see Fig. 2, PET- first DSC scanning at approx. 90 °C) obstructing the real glass transition and leading to erroneous results. The researchers have also reported that such shift moves to lower temperatures with either increasing moisture or decreasing temperature of polymer's exposure.

For control (empty) PET bottles the T_g values slightly declined towards the end of the storage period (Fig. 3) independent of the storage conditions, though of no statistically significant differences. Also, no significant difference were reported between control and oil-exposed PET samples placed at any storage temperature (Piergiovanni et al., 1995).

The T_m of control and oil-exposed PET bottles decreased towards the end of the storage period as shown in Fig. 3a & b.

As indicated, T_m did not differ significantly for any temperatures between the control and the oil exposed PET. For the same temperature there was a significant change of the T_m after 12 months of storage compared to the original value for either the oil exposed or the control bottles. Over all, T_g and T_m parameters were statistically more influenced by temperature rather than light.

The percent crystallinity was evaluated for both the first and second DSC scans. Values of the % crystallinity for PET (X_{DSC}), shown in Fig. 4, were calculated using:

$$X_{DSC} = \Delta H_{exp}/\Delta H_o, \quad (2)$$

where ΔH_o for PET is 117.6 J/g.

Values of the % crystallinity recorded during the second scan were about 10% lower compared to the first one, most likely due to the possibility that heating during the first scanning may have eliminated

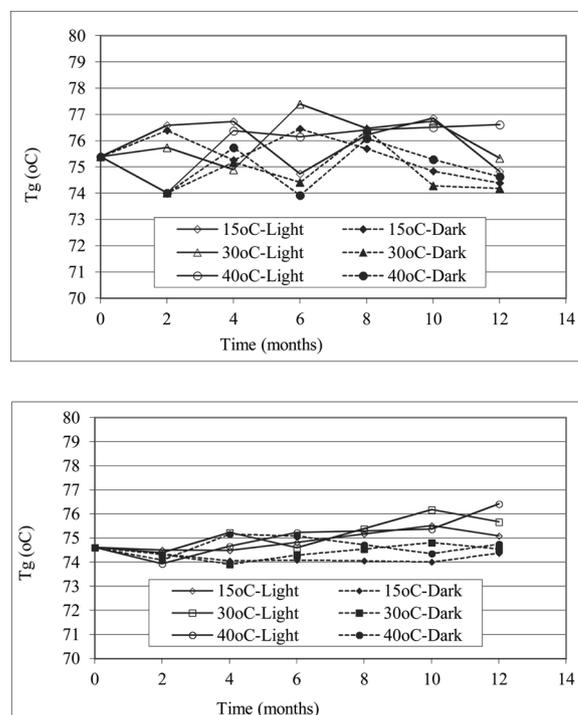


Fig. 3. T_g values for PET control (Fig. 3a) and olive oil-exposed (Fig. 3b) bottles recorded during the second DSC scan. Bottles were kept at 15, 30 and 40 °C in light or dark and light.

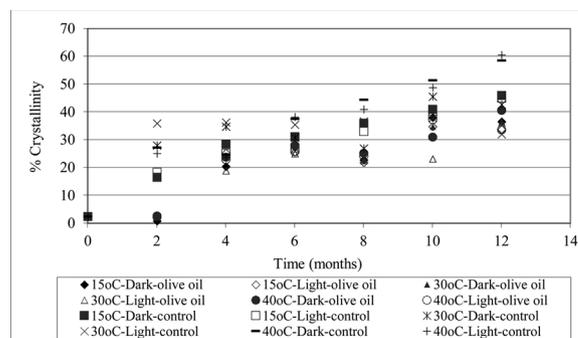


Fig. 4. Changes of % crystallinity for olive oil exposed and control PET bottles during storage. Bottles were kept at 15, 30 and 40 °C in light or dark.

Table 1

Infrared spectroscopic ν (cm^{-1}) data, for PET bottles, of characteristic absorbcency frequencies at the infrared region for the molecules involved in the PET polymers structure.

Polymer type	FTIR (cm^{-1})
PET	2969, 1713, 1409, 1341, 1244, 1099, 1020, 873, 727

some of the crystalline areas or pseudo-crystallites, while fast cooling allowed little time for crystallization to occur.

An increased crystallinity was observed for PET bottles stored at 40 °C (Fig. 4). That could probably be explained by the possibility of an increased molecular mobility experienced at increased temperatures, along with a rearranging of molecules to the crystalline state, encouraging crystallization in the solid glassy state below T_g .

Moreover, the crystallinity % increased with time. The increase was higher for the control, as compared to the bottles filled with olive oil. A more significant increase was reported for the bottles stored at 40 °C, while light did not seem to influence the results. The statistical analysis

Table 2

Olive oil-related FTIR absorbency frequencies, functional groups responsible and their mode of vibration (Guillén & Cabo, 1997; Guillén & Cabo 1999).

Frequency (cm ⁻¹)	Functional group	Mode of vibration
3648	-C=O (ester)	Overtone
3025	=C-H (<i>trans</i> -)	Stretching
3006	=C-H (<i>cis</i> -)	Stretching
2953	-C-H (CH ₃)	Stretching (asym)
2924	-C-H (CH ₂)	Stretching (asym)
2853	-C-H (CH ₂)	Stretching (sym)
2730	-C=O (ester)	Fermi resonance
2677	-C=O (ester)	Fermi resonance
1746	-C=O (ester)	Stretching
1711	-C=O (acid)	Stretching
1654	-C=C- (<i>cis</i> -)	Stretching
1648	-C=C- (<i>cis</i> -)	Stretching

showed that the %crystallinity of the control PET bottles after 12 months at 15 °C differed significantly from the %crystallinity of the bottles stored at 40 °C during the same time period. PET bottles after 12 months at 30 °C had a % crystallinity significantly different to the bottles stored at 40 °C. Olive oil exposed PET containers did not differ among any of the storage temperatures used in this study.

3.3. FTIR

FTIR analysis of PET based on their characteristic peak frequencies and the bands of olive oil are respectively given in Table 1 and Table 2.

A significant overlapping of the common absorption bands for the two materials, did not allow a more clear FTIR spectra for the olive oil related absorption bands. Thus, it was concluded that only humidity, which had penetrated into the polymers, could be recorded. Moreover, as indicated by Vijayakumar and Rajakumar (2012), neither an investigation of the carbonyl stretching amorphous band at 1727 cm⁻¹ nor the crystalline band at 1717 cm⁻¹ by TA-FTIR were possible to be recorded, due to overlapping effects.

In general, PET samples did not show alterations of the FTIR spectrum bands under these storage conditions. Also, migration of compounds that was reported when subjected to higher temperatures, was not important given the storage conditions for this study (Castle, Mayo, Crews, & Gilbert, 1989; Reeves, 1997).

In this study, the PET bottles containing olive oil seem to have absorbed less water compared to the control ones. That may be concluded via the changes at the 5240 cm⁻¹ wavelength, a characteristic

absorption band by the water molecules. The control PET bottles absorbed about 35% more water than the oil-exposed bottles at any temperature and light conditions after 12 months of storage at 40 °C (Fig. 5). Water sorbed by the polymer increased with the storage temperature (Fig. 6), in accordance to the previously reported work of Jabarin and Lofgren (1986). According to Jabarin and Lofgren (1986), the physical properties of PET were significantly influenced by the relative humidity sorbed when exposed to various environments (temperature, RH) during time.

3.4. VIS-light transmittance

In general, PET bottles were more opaque to UV-region light allowing almost no radiation of wavelengths below 340 nm to pass through (Fig. 7).

PET bottles did not show any changes in the light transmitted through them during storage under any of the studied conditions and time.

3.5. Colour

For this study, the L* value, that indicates lightness, the a* value, that moves from red (+a*) to green (-a*) and the b* value, for yellow (+b*) to blue (-b*), were reported since they can provide a quite clear description of the color changes over time, being affected by light and/or radiation (El-Zaher, 1999; Grassie, 1972). No clear colour changes were recorded for control or oil-exposed PET bottles during the storage time.

4. Conclusions

This work assessed the influence of olive oil, temperature, light, and humidity, on the thermal properties, crystallinity, color, sorption and oxygen and light barrier properties of PET packaging materials, during a one-year storage time. In general, the polymeric materials' properties, and consequently their relevant performance during food preservation, undergo certain changes. Polymeric materials in contact to olive oil seemed to have distinct different behavior concerning the thermal properties of the polymers, the permeability values during time, the antagonistic sorption of environmental (RH) and product's constituents and potential degradation phenomena, as compared to control materials.

This work, solely supports the opinion that the shelf life estimations of the oils, and in particular of the olive oil, might be erroneously

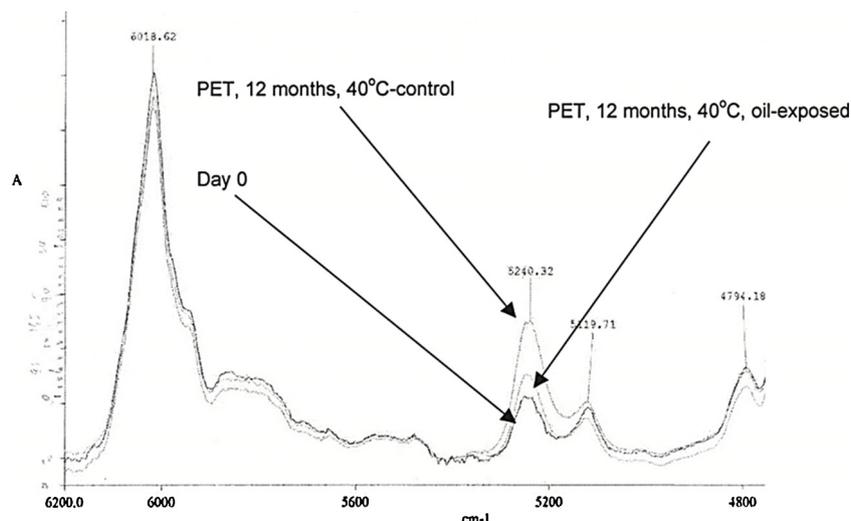


Fig. 5. FTIR analysis of the control PET bottles stored at 15, 30 and 40 °C. The height of the absorption band at 5247 cm⁻¹ is related to the amount of water sorbed by the polymer after 12 months of storage.

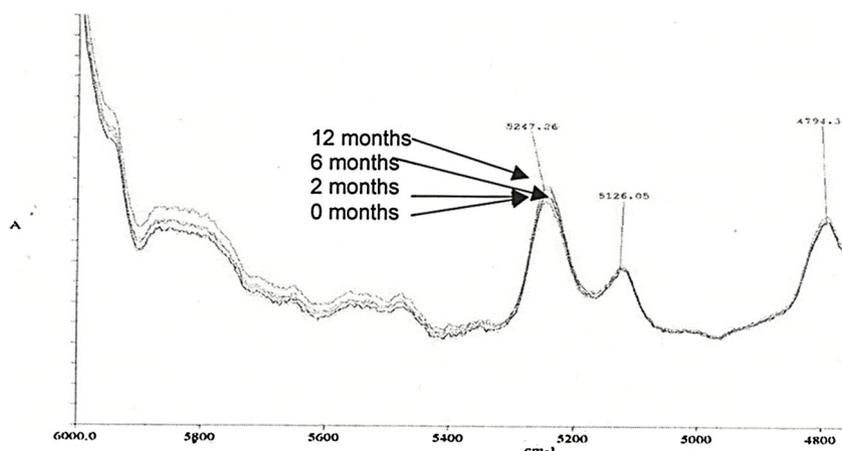


Fig. 6. FTIR analysis of the oil exposed PET bottles. The height of the absorption band at 5247 cm^{-1} is related to the amount of water sorbed by the polymer stored at $15\text{ }^{\circ}\text{C}$ for 0, 2, 6 and 12 months.

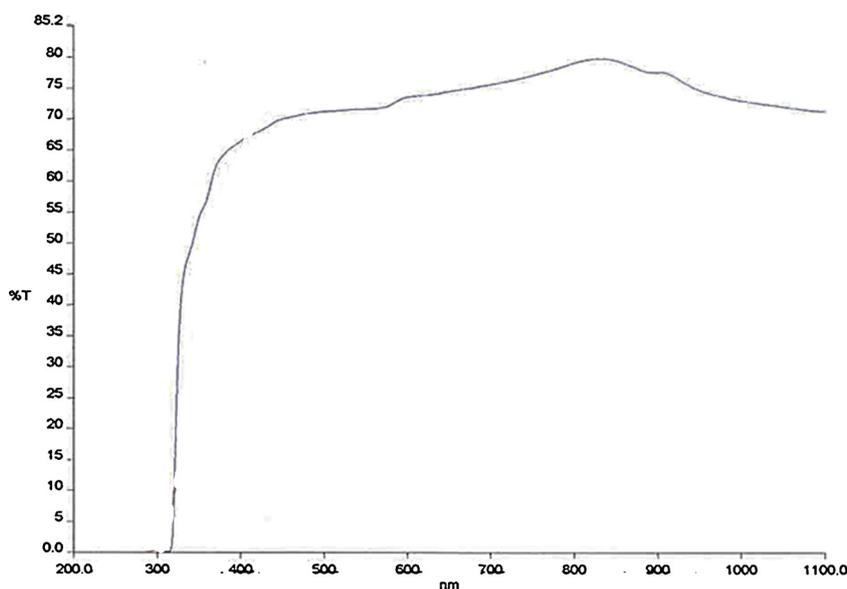


Fig. 7. PET light transmission % for control bottles.

estimated via models that consider the permeability as a constant factor. Although, the significance of the storage environment has been broadly stated for the olive oil, this work takes a step further in the area of food-packaging –environment system, by suggesting the impact of the alterations within the polymers during a realistic period of storage, throughout which, the package is in contact with the product. Furthermore, in order to precisely assess the overall performance of the food-packaging-environment system in its totality, the visual appearance of a package should be also validated, assisting in the selection of the most suitable materials and storage conditions. Conclusively, the shelf life of olive oil strongly depends on the oxygen barrier properties of the packaging materials, only to be regulated by elevated temperatures.

This study clearly showed that various oil-package interactions occur during the PET-oil contact that may affect the performance and barrier properties of the PET material. According to that reported data, we may suggest that for a more accurate evaluation of shelf-life, the implementation of the oxygen transmission rates should be considered over time. That approach, should be quite more representative of the actual oxidation phenomena under a given set of conditions and prolonged time storage.

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