



*Research article*

## **Light protection performance of wrapping films to prevent the photo-oxidation of extra virgin olive oil during storage in glass bottles**

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**Abstract:** Packaging material plays an important role in minimizing food waste in the supply chain. This study evaluated the light-protective effects of wrapping films as a secondary packaging material on the deterioration of extra virgin olive oil after exposure to sunlight for 5 weeks. Milky white polyethylene terephthalate (MW-PET) and aluminum oxide vapor-deposited polyethylene terephthalate (AV-PET) were used on bottles of different transparency (clear or amber glass) as the primary packaging material. Wrapping the bottles with either PET film resulted in protection against fatty acid release and secondary oxidation of the oils upon exposure to sunlight, but no protection against the primary oxidation of the oils. Although sunlight exposure induced the degradation of minor components, including chlorophyll and carotenoid pigments, in oils stored in clear glass bottles with no film, in oils stored in clear and amber glass bottles with either type of PET film, these compounds were partially protected from degradation during storage. In addition, sunlight exposure induced a decrease in E2-hexenal, which has a positive olive aroma in oils stored in clear glass bottles with no film, but this decrease was not observed to the same extent in oils stored in clear and amber glass bottles with either PET film. The light-protective effects of the AV-PET film were significantly higher than those of the MW-PET film. These results showed that secondary packaging with these PET films provided more effective protection against sunlight-induced oxidative deterioration of extra virgin olive oil during storage in primary materials, such as glass bottles, compared with storage in glass bottles with no secondary packaging.

**Keywords:** olive oil; oxidation; packing film; storage; glass bottle; light protection; food waste

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## 1. Introduction

Edible oils are usually packed in transparent or colored glass packaging or plastic packaging. There are often situations in which vegetable oils, including olive oil, are exposed to direct sunlight or artificial light in the systems of distribution and retail operations. Light, oxygen, humidity, and temperature are some of the extrinsic factors that adversely affect the composition and quality of fats and oils during and after processing [1]. In particular, light can act as an initiator of oxidative reactions that lead to the deterioration of oils [2,3]. Although fats are not able to absorb visible light, oxidation can be induced by light that has been absorbed by chlorophyll pigments in the oil [4–6]. The light-induced formation of free radicals leads to the auto-oxidation of fats [7].

Because of its nutritional properties, the consumption of extra virgin olive oil (EVOO) plays a vital role in preventing cardiovascular disease [8]. Therefore, this oil is very popular around the world and is globally available in different packages with attractive designs made of transparent packaging materials. Shopkeepers and retailers often keep oils packed in such containers outside their shops, thus directly exposing the containers to sunlight, for the purposes of advertising or because of insufficient space in their shops. In households, oils may also be exposed to daylight on a sunny windowsill in the home kitchen. Such lack of good practice in the shipping, storage, and handling of oils can initiate photooxidation. Interaction of the oils with the environment can result in rancidity, which reduces the quality of the oils resulting in the loss of the economic value and eventually disposal of the oils, in the distribution (i.e., supermarkets and retailers where food waste, including unsold products, is generated) and consumption (i.e., restaurants and households) stages of the food supply chain.

Although packaging should be attractive for consumers, it also plays a crucial role in the protection from light to maintain the quality of EVOO throughout the commercial life of the product. Because glass materials provide not only a good barrier against gases and moisture but also a low transmittance of light in the UV range, glass is a suitable material for food contact for packaging of oils, and provides protection during storage [9,10]. Limiting the transparency of the glass packaging can provide protection against oil deterioration from sunlight; however, the protective effect is insufficient because the oils are still exposed to some visible light [9,11,12]. Therefore, further research is important to evaluate the protective effects of packaging materials on the extent of oxidative degradation produced by sunlight that affects the quality of oils, in an attempt to reduce the amount of unsold oil products. Few studies have shown that multilayered plastic-coated paperboard aluminum foil laminate packaging proved could preserve the overall quality of EVOO than glass packaging during storage under light conditions [14–17].

Previous studies have only evaluated primary packaging materials that are in direct contact with the oil [9–12]. Secondary packaging materials, including wrapping features that are not in contact with the oil, may be useful to realize light protection. However, little information is available regarding the characteristics of such extra packaging materials on preventing sunlight exposure. Aluminum foil acts as an excellent secondary packaging material, whereas it has advantages, such as its high cost and limited structural strength [12,18]. Polyethylene terephthalate (PET) is used widely to be as a commodity polymer in the food packaging. PET films effectively block oxygen, water vapor, and light, and have excellent adhesive promotion, thickness variation, and weather resistance properties, which are suitable for a secondary packing material [19,20]. In particular, as metallized polymer films provide improved performance in terms of the barrier properties, aluminum oxide barrier coatings on polymer films are more tear resistant, flexible and lighter

than aluminum foil [21,22]. The metallized PET films can be made of a smaller amount of the aluminum that would have been needed for an aluminum foil. Therefore, the metallized PET film become a more interesting as a cost-effective and more environmentally friendly alternative to aluminum for food packaging applications. The objective of the present study was to evaluate the protective effects of PET wrapping films that effectively block light on the photooxidation of EVOO in different transparent glass bottles during storage with exposure to sunlight.

## 2. Materials and methods

### 2.1. Materials

EVOO (Exp. Date: 07/2022; Shodoshima Healthyland Co., Ltd., Kagawa, Japan) and medium-chain triglyceride (MCT) oil (Nisshin OilliO Group, Ltd., Tokyo, Japan) were purchased in a market. The fatty acid composition of the EVOO was: palmitic (C16:0; 13.8%), palmitoleic (C16:1; 1.4%), stearic (C18:0; 1.8%), oleic (C18:1n-9; 69.8%), cis-vaccenic (C18:1n-7; 3.4%), linoleic (C18:2n-6; 8.0%),  $\alpha$ -linolenic (C18:3n-3; 1.0%), arachidic (C20:0; 0.3%), eicosenoic (C20:1n-9; 0.3%), and behenic (C22:0; 0.1%). The amount of  $\alpha$ -tocopherol in the EVOO was 24.6 mg per 100 g of oil. E2-hexenal (purity > 97%) was purchased from FUJIFILM Wako Pure Chemical Co. (Osaka, Japan).

### 2.2. Sunlight storage conditions

According to packing of 200 grams of EVOO into 250 mL glass bottle in the olive oil retail market, the experiments were conducted based on the ratio between oil and air in the bottle: three samples (40 g) of oil for each storage condition were weighed into 50 mL clear glass bottles and 50 mL amber glass bottles with the headspace occupied by air. To shield some of the samples from sunlight, these samples in the clear and amber glass bottles were covered with milky white PET (MW-PET) film (#3-8016-01; Tokyo Film Service Co., Ltd., Tokyo, Japan) that has excellent electrical, mechanical, heat-resistant, and chemical properties, and milky white-colored, aluminum oxide vapor-deposited PET (AV-PET) film (#50H10; AS ONE Co., Osaka, Japan). Three samples in clear glass bottles wrapped with aluminum foil were used as a control to be completely protected from sunlight [12,13]. To simulate typical light exposure at the consumer level, the oil samples were then placed on a south-facing windowsill in the laboratory at a room temperature of  $22 \pm 3$  °C [11,23] for a period of 5 weeks through February to March 2021 (the mean value of cumulative sunshine hours per week in Shodoshima was  $48.0 \pm 8.3$  h, which was based on the weather data provided by the Japan Meteorological Agency). In terms of consumer behavior for the initial amount of oil, for each set of experiments an equal amount of oil (4 g) was taken from the same bottle and analyzed weekly.

### 2.3. Analytical procedures

The free acidity (FA), peroxide value (PV), and absorption value at 270 nm ( $K_{270}$ ) of the oil samples were measured using an OxiTester (CDR; Ginestra Fiorentina, Italy) [12,24,25]. FA values are expressed as the percentage content of the free fatty acids in oil. PV states the milliequivalents of primary peroxide oxygen combined in a kilogram of oil. The  $K_{270}$  value is coefficient of extinction to measure the state of secondary oxidation of the oil. Samples of the oils were added to prefilled cuvettes for analysis. The volumes of oil used were: 2.5  $\mu$ L for measuring FA (%), 0.5–2.5  $\mu$ L for

PV (meqO<sub>2</sub>/kg), 10 µL for K<sub>270</sub>. The fatty acid composition and the α-tocopherol content of the EVOO were determined by Japan Food Research Laboratories (Tokyo, Japan). The content of chlorophyll and carotenoid pigments, reported as mg/kg of oil, was determined using a UV-1800 spectrophotometer (Shimadzu Co., Kyoto, Japan) following a slightly modified method to that described previously [12,26]. A hundred micrograms of the oil samples were dissolved in 1 mL of isoctane, then the absorption was recorded at 670 nm for chlorophyll and 470 nm for carotenoid pigments. The content was calculated using the following equation:

$$\text{Chlorophyll pigments (mg/kg)} = (A_{670} \times 10^6) / (613 \times 100 \times d) \quad (1)$$

$$\text{Carotenoid pigments (mg/kg)} = (A_{470} \times 10^6) / (2000 \times 100 \times d) \quad (2)$$

Where  $A$  is the absorption and  $d$  is the path length of the cell (1 cm).

#### 2.4. Flash gas chromatography electronic nose analysis of E2-hexenal

The volatile organic compounds in the headspace of the oil samples were analyzed using a HERACLES II electronic nose (Alpha MOS, Toulouse, France) [12,27]. The HERACLES II was equipped with two identical gas chromatographic columns working in parallel: a non-polar column (MXT-5: 10 m length × 180 µm diameter) and a polar column (MXT-WAX: 10 m length × 180 µm diameter), which produced two chromatograms simultaneously. The HERACLES II was also equipped with an HS 100 auto-sampler (CTC Analysis AG, Zwingen, Switzerland) to automate the sample incubation and injection. An alkane mixture (from *n*-hexane to *n*-hexadecane) was used to convert retention times into Kovats indices for calibration. For analysis, an aliquot of oil (2.0 g) was placed in a 20 mL vial, then sealed with a magnetic cap. The vial was placed in the auto-sampler, which was subsequently placed in the shaker oven of the HERACLES and incubated for 15 min at 60 °C with shaking at 500 rpm. A syringe was used to sample 5 mL of the headspace for injection into the gas chromatograph. The oven temperature was initially set at 40 °C (held for 10 s), then increased to 250 °C at 1.5 °C/s and held for 60 s. The total separation time was 120 s. Hydrogen gas was used as the carrier gas. The data were acquired and processed using AlphaSoft software v2020 (Alpha MOS). The AroChemBase module (Alpha MOS) was used to identify the volatile compounds. When using the MXT-5 and MXT-Wax columns, E2-hexenal eluted at approximately 53 s.

#### 2.5. Quantification of E2-hexenal

The standard curve for E2-hexenal was used to determine their concentrations in the oil samples [12,27]. Different concentrations of E2-hexenal were prepared in an MCT oil then subjected to flash gas chromatography electronic nose analysis. The high value of the coefficient of determination ( $R^2 = 0.999$ ) indicated that the standard curves allowed the quantification of E2-hexenal in the oils with a high level of accuracy.

#### 2.6. Statistical analysis

Data are presented as means ± standard deviation from three replicates. The data were analyzed by one-way analysis of variance followed by the Tukey-Kramer test in Microsoft Excel. Differences between mean values were considered statistically significant at  $p < 0.05$ . Linear Pearson correlation

coefficient (*R*-Pearson) was applied to evaluate the existence of bivariate correlations within the EVOO's physicochemical parameters.

### 3. Results and discussion

#### 3.1. Effect of the wrapping films on EVOO chemical parameters during storage in glass bottles under sunlight conditions

At the packaging, the test olive oil could be labeled as EVOO, which is the highest grade of olive oil, based on the physicochemical parameters (Table 1), which showed values within the legal limits for EVOO according to the International Olive Council (IOC) regulations [28]. The initial values of the chlorophyll and carotenoid content in the oil are also shown in Table 1.

**Table 1.** Chemical characterization of EVOO at the time of packaging.

Analytical parameters	At the packaging	The IOC regulation
Free acidity (%)	0.04 ± 0.02	≤0.80
Peroxide value (meqO <sub>2</sub> /kg)	4.8 ± 0.3	≤20.0
K <sub>270</sub>	0.074 ± 0.016	≤0.22
Chlorophylls (mg/kg)	2.46 ± 0.03	–
Carotenoids (mg/kg)	1.72 ± 0.02	–

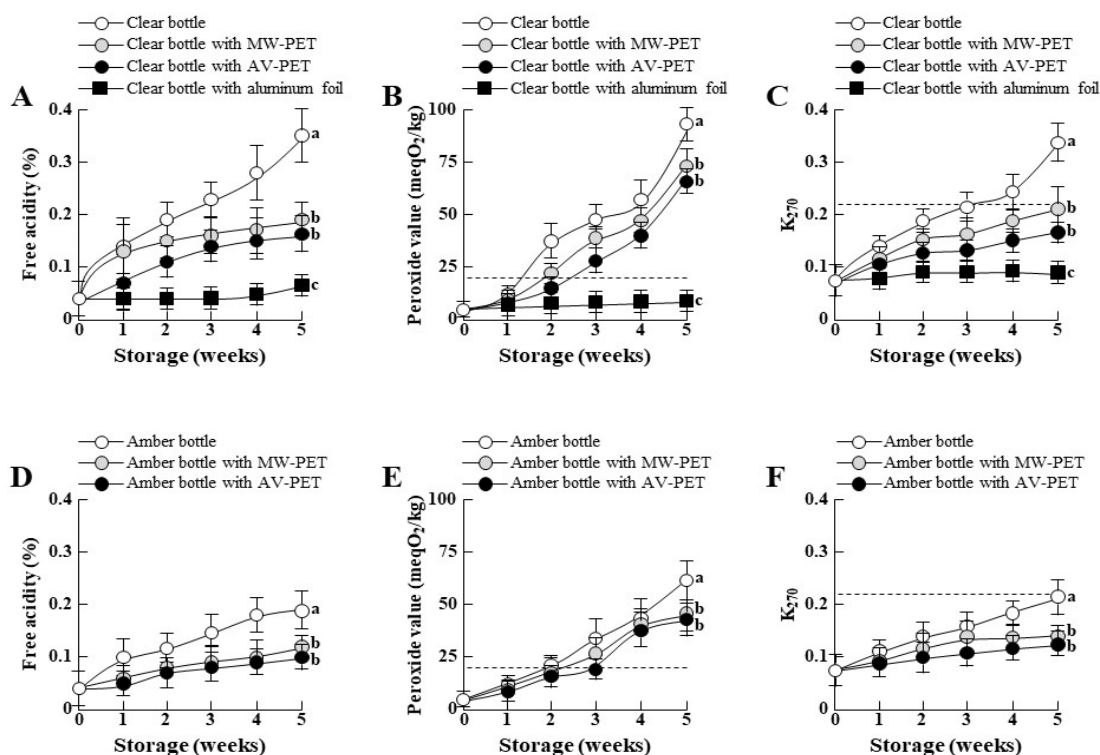
EVOOs stored at different glass bottle conditions showed similar time evolution trends were observed for the chemical quality parameters during storage time ( $0.81 < R\text{-Pearson} < 0.99$ ). The FA values of the EVOOs stored in clear glass bottles with different wrapping films (none/MW-PET/AV-PET) were positively correlated with PV and K<sub>270</sub> values ( $0.65 < R\text{-Pearson} < 0.93$ ). It was also found a significant positive correlation of the FA values of the EVOOs stored in amber glass bottles without or with different wrapping films (MW-PET/AV-PET) with PV and K<sub>270</sub> values ( $0.82 < R\text{-Pearson} < 0.91$ ). Thus, EVOOs with greater FA had higher PV and K<sub>270</sub> levels.

The FA of olive oil results from the hydrolysis of triacylglycerols. The FA values of EVOO samples stored under different packaging conditions with exposure to sunlight are shown in Figure 1A and 1D. As previously reported [12], the FA values of EVOO stored in clear glass bottles with no film increased gradually as the exposure time to sunlight increased but did not exceed the 0.80% maximum for EVOO [28]. Aluminum foil wrapping could prevent the light-induced lipid oxidation in the oil. For EVOO stored in clear glass bottles wrapped with either MW-PET film or AV-PET film, a slight increase in the FA value was observed after exposure to sunlight for 5 weeks, which was significantly smaller than that for EVOO stored in clear glass bottles with no film. The FA values of EVOO stored in amber glass bottles wrapped with either film was significantly smaller than that in amber glass bottles with no film. In both of the clear and amber glass bottles, wrapping with AV-PET film tended to give EVOO with a smaller FA value than wrapping with MW-PET film. These observations suggested that wrapping with these PET films can protect against the hydrolysis of triacylglycerols upon exposure to sunlight.

The PV increases as a consequence of the action of primary oxidation and is widely used as an indicator of fat oxidation, and is often used for monitoring peroxide formation during the initial stages of lipid oxidation (Figure 1B and 1E). The PVs of EVOO stored in clear glass bottles with

aluminum foil did not increase significantly during storage. The oil samples stored in clear and amber glass bottles with no film exhibited higher PVs after exposure to sunlight for 2 weeks, which exceeded the limit in the standard; therefore, these oils could no longer be classed as EVOO. In contrast, the PVs of oils stored in clear glass bottles with either film and in amber glass bottles with MW-PET film did not exceed the 20.0 meqO<sub>2</sub>/kg standard for EVOO after exposure to sunlight for 2 weeks [28]. The PVs of the oils stored in amber glass bottles with AV-PET film remained below the standard value for EVOO after exposure to sunlight for 3 weeks. These observations suggested that these PET films can partially protect against the primary oxidation of EVOO upon exposure to sunlight.

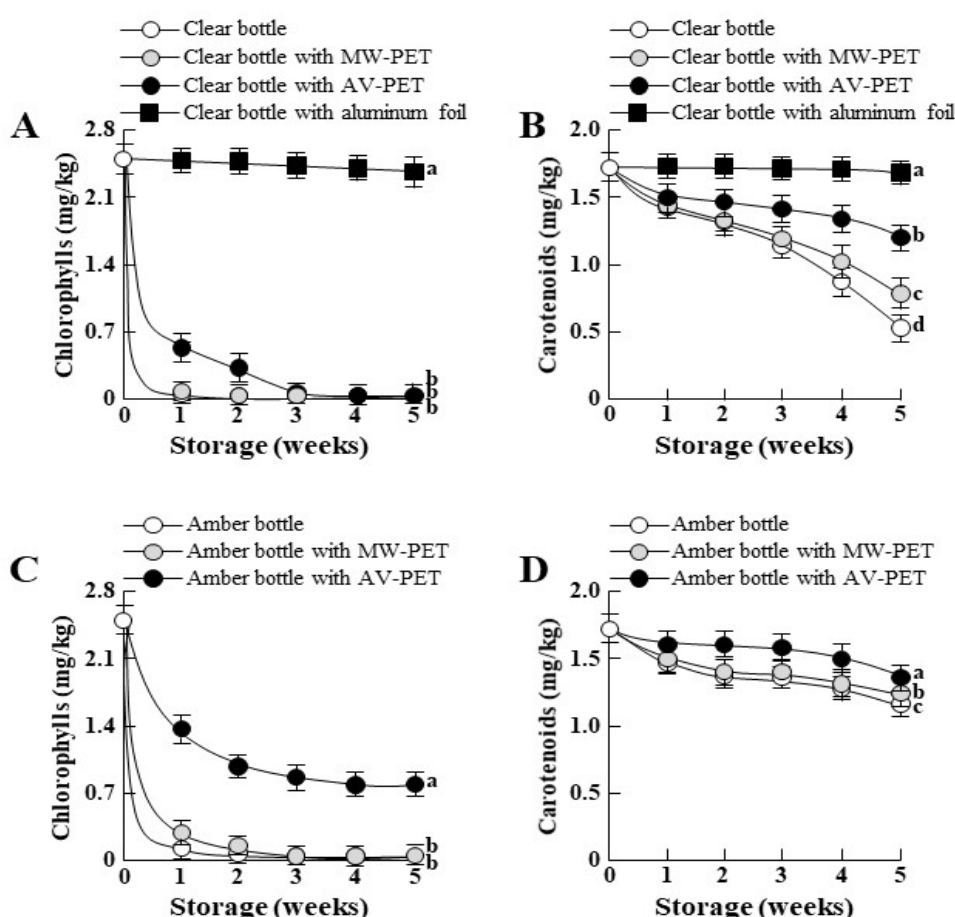
It is possible to verify the degree of olive oil oxidation by assessing the K<sub>270</sub> value that indicates the presence of carbonyl compounds, which correlates with the presence of secondary oxidation compounds [29]. Changes in the K<sub>270</sub> value of EVOO stored in the different packaging materials were measured during storage with exposure to sunlight (Figure 1C and 1F). With increasing storage time, exposure to sunlight induced a progressive increase in the K<sub>270</sub> value for EVOO stored in clear glass bottles with no film. The K<sub>270</sub> values for the oils stored in clear and amber glass bottles with no film exceeded the 0.22 limit in the standard for EVOO [28] after exposure to sunlight for 3 and 5 weeks, respectively. In contrast, the K<sub>270</sub> value for EVOO stored in clear glass bottles with the films and in amber glass bottles with and without the films remained within the quality range for EVOO throughout the storage period. The K<sub>270</sub> value of EVOO stored in clear glass bottles with aluminum foil did not increase significantly during storage. These observations suggested that these PET films can protect against the secondary oxidation of EVOO upon exposure to sunlight.



**Figure 1.** Changes in free acidity (A, D), peroxide value (B, E), and K<sub>270</sub> value (C, F) of EVOO during storage with exposure to sunlight. Dashed lines: legal thresholds (the IOC regulation). <sup>a-c</sup>For the 5 week values, the mean values with different letters were significantly different ( $p < 0.05$ ).

### 3.2. Effect of wrapping films on EVOO chlorophyll and carotenoid pigments during storage in glass bottles under sunlight conditions

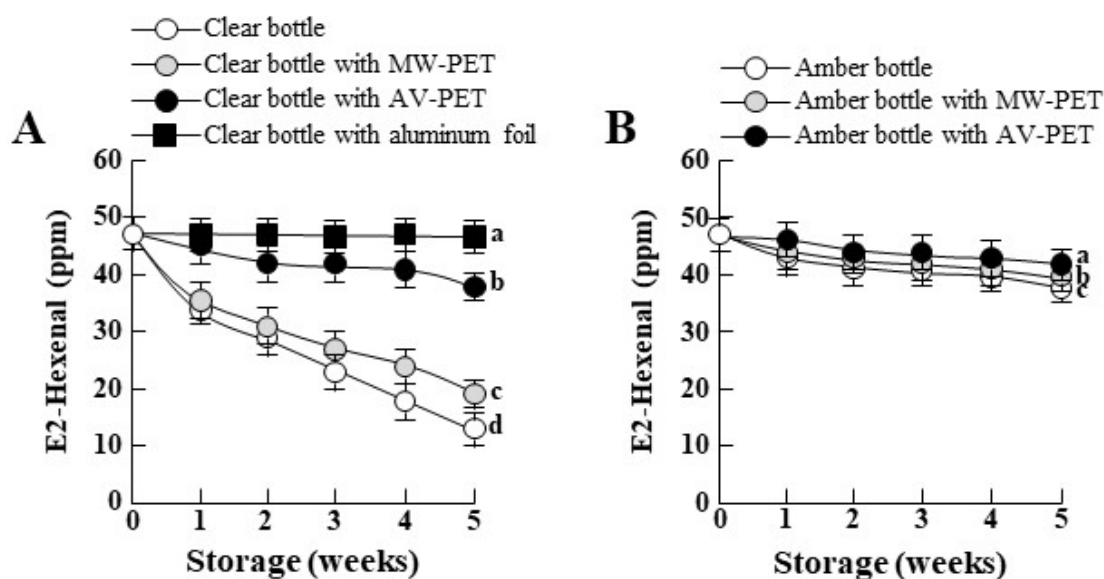
The color of EVOO is mainly related to the presence of chlorophyll and carotenoid pigments, which cause green and yellow coloration, respectively [30]. These pigments influence consumer acceptance of the oil and the oil stability. Figure 2 shows the changes in the levels of these pigments in EVOO stored in clear and amber glass bottles with the different films during sunlight exposure. Both pigments were stable in EVOO stored in clear glass bottles with aluminum foil (Figure 2A and 2B). Sunlight exposure resulted in severe loss of chlorophyll compounds from EVOO stored in clear glass bottles with MW-PET film, as well as in bottles with no film, and these compounds were completely degraded after sunlight exposure for 1 week (Figure 2A). The rate of decrease with the AV-PET film was slightly slower than that with the MW-PET film and the chlorophyll compounds were completely degraded after sunlight exposure for 3 weeks. EVOO stored in amber glass bottles showed the same trends as observed for the clear glass bottles regarding the chlorophyll content (Figure 2C). The chlorophyll content in EVOO stored in amber glass bottles with AV-PET film was retained partially after sunlight exposure for 5 weeks (Figure 2C).



**Figure 2.** Changes in chlorophyll (A, C) and carotenoid (B, D) levels in EVOO during storage with exposure to sunlight. <sup>a-d</sup>For the 5 week values, the mean values with different letters were significantly different ( $p < 0.05$ ).

### 3.3 Effect of wrapping films on EVOO volatile compounds during storage in glass bottles under sunlight conditions

E2-hexenal contributes considerably to the aroma of olive oil and is related to the positive sensory characteristics of almond and green olive fruits [31,32]. Figure 3 shows the changes in the E2-hexenal content in EVOO stored in glass bottles during sunlight exposure. The E2-hexenal content in EVOO stored in clear glass bottles with aluminum foil remained at the initial level throughout the storage period (Figure 3A). The E2-hexenal content in EVOO in clear glass bottles with AV-PET film also remained high during storage. In contrast, the E2-hexenal content in EVOO stored in clear glass bottles with no film, and wrapped with MW-PET film, decreased as the storage time increased, although the E2-hexenal content in clear glass bottles with MW-PET film was significantly higher than that in bottles with no film (Figure 3A). EVOO stored in amber glass bottles with no film retained the E2-hexenal content at a high level (Figure 3B). The E2-hexenal content in EVOO stored in amber glass bottles with either MW-PET film or AV-PET film was slightly higher than that with no film. These observations suggested that wrapping glass bottles with either type of PET film can partially maintain the olive oil aroma in EVOO during storage with exposure to sunlight.



**Figure 3.** Changes in the E2-hexenal content of EVOO stored in clear (A) and amber (B) glass bottles with different wrapping materials during storage under conditions of sunlight. <sup>a-d</sup>Mean values after storage for 5 weeks with different letters were significantly different ( $p < 0.05$ ).

## 4. Conclusions

Based on the data obtained in this study, the quality evolution of EVOO during storage with exposure to sunlight is influenced by wrapping with the secondary packaging films. Wrapping glass bottles with MW-PET or AV-PET film did not provide sufficient protection against the primary oxidation of EVOO, as primary peroxide formation was rapidly elevated, during storage under



conditions of sunlight, but provided protection against the secondary oxidation of EVOO and showed a low level of free fatty acids in EVOO. The sunlight-induced degradation of carotenoid pigments in EVOO was partially prevented by storage in glass bottles wrapped with either type of PET film, but no protection against the degradation of chlorophyll compounds was observed. Wrapping with either type of PET film partially protected against the decrease in a positive olive aroma of EVOO stored in clear glass bottles observed upon exposure to sunlight. Notably, the light protection performance of the AV-PET film was superior to the MW-PET film for all the oil quality categories tested. These findings will help to select appropriate primary and secondary packaging materials, ensuring more effective protection from light, which together with bottling procedures that maintain the quality of the oil for as long as possible, may lead to an increase in the shelf life of the oil. Eventually, it is expected that these light-barrier packaging materials will have an impact on reducing food waste in the olive oil supply chain.

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### Conflict of interest

The author declares no conflicts of interest.

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