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RESEARCH ARTICLE

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Light barrier properties of extra virgin olive oil (EVOO) glass bottles—Effect of glass colour and decoration

S.B.M. Jaime[1] | P.F.J. Bócoli[1] | K. Miguel[2] | E.A.H. Ferreira[3] | R.M.V. Alves[1]

[1] Packaging Technology Centre, Institute of Food Technology, Campinas, SP, Brazil

[2] Faculty of Food Engineering, University of Campinas, SP, Brazil

[3] Science and Quality of Food Centre, Institute of Food Technology, Campinas, SP, Brazil

Correspondence
S. B. M. Jaime, Packaging Technology Centre, Institute of Food Technology, Campinas, SP Brazil.
Email: sandra@ital.sp.gov.br

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The aim of this study was to evaluate the quality of some glass bottles used for extra virgin olive oil (EVOO) related to light barrier properties, including clear and coloured glass in different colour intensities, glass with shrink labels, and external ceramic decoration. Some of these glass bottles available on the Brazilian market were also evaluated for closure integrity and barrier to oxygen, headspace gas atmosphere, and the quality of the product according to legislation. An increase in light barrier properties was observed for the darkest glass bottle that depends on the pigment quantity and the wall thickness. A total light barrier was obtained for glass with black shrink labels and external ceramic decoration. Closure systems used for the analysed packaging showed good integrity and low oxygen permeability, and the inertness of headspace atmosphere ensured a lower availability of oxygen for product oxidation. In general, the products met the requirements imposed in the legislation.

KEYWORDS

colour influence, extra virgin olive oil (EVOO), glass bottles, light transmittance

1 | INTRODUCTION

Over the last decade, an increase in the appreciation for extra virgin olive oil (EVOO) among Brazilian consumers has been observed due to various factors including more per-capita purchasing power, and especially benefits offered by a Mediterranean diet.[1,2] Some regions in Brazil now have more olive tree plantations for commercial purposes, but the country is still dependent on olive and olive oil imports, mainly supplied by the European Union.[1]

EVOO is considered the best olive oil for its organoleptic, nutritional and sensory qualities, stability, and chemical composition.[3-6] It is made by crushing olives and extracting the oil by centrifugation or pressure without chemical or industrial refining. The oil quality and taste may be influenced by the olive variety and its maturation stage, the harvesting period, and the storage condition of the final product.[7]

In Brazil, olive oil characteristics, their quality, and criteria for a correct classification of commercial olive oil have been approved by Legislation N° 1/12, published by the National Agricultural Agency,[8] which complies with international legislations concerning olive oil.[9]

Similar to other products, olive oil is produced in a limited period but is consumed throughout the year. Therefore, storage conditions and the type of packaging are important parameters to prevent degradation of the product and allow for its worldwide distribution and retention of quality for an extended period from production to consumption.[5,10,11]

Many factors affect the quality of the storage of olive oil, and oxidation leading to rancidity is the most important cause of oil deterioration after microbial spoilage.[3,6,10,11] Rancidity can be caused by auto-oxidation and photo-oxidation. Auto-oxidation is the deterioration of EVOO in the absence of light due to free radical triplet oxygen. When the product is exposed to light, photo-oxidation occurs through natural photosensitizers found in the oil, the chlorophyll.[3,6] Chlorophylls is the main photosensitizer in EVOO which, in the presence of light, can accelerate the oxidation level of the product at a given storage time.[3] Chlorophyll also plays a significant role in the initiation of lipid oxidation.[11]

Some studies also observed that the amounts of chlorophylls drastically decreased after few months of storing EVOO in clear glass bottles under light.[7,12,13] Chlorophyll content in EVOO filled in coloured (amber) glass bottles remained constant for 6 months even under light storage conditions.[7] The visible light absorbance spectrum of chlorophyll creates peaks in the range of 400 to 500 nm (blue region) and 600 to 700 nm (red region),[14] and therefore, it is of most importance to know previously about light protection of the packaging material in order to reduce or avoid photo-oxidation of the product.

Some studies have shown that the quality of olive oil is influenced by the type of packaging material (glass, PET, PP, PET with oxygen scavenger, tin, PVC, etc.), storage conditions (light, temperature), and time.[3,5-7,11] Packaging can directly influence the quality of olive oil by protecting the product from both oxygen and light barriers.[6] According to a study conducted by Kanavouras and Coutelieris,[11] even short-term exposure of the oil to light should be avoided as light could significantly stimulate oxidative degradations, especially when exposed to high temperatures and in the presence of oxygen.

Cecchi[3] observed that chlorophylls in EVOO decreased very quickly in the presence of light when packaged in transparent PET bottles stored under diffuse light conditions at room temperature, even in transparent PET bottles with oxygen scavengers. The presence of oxygen scavengers dispersed in PET bottles demonstrated lower oxidation level for EVOO in dark storage as it provides a barrier to O_2 diffusion from the atmosphere, but not against light.[15] Even different coloured PET bottles (clear, green, orange—transparent plastic, white and blue—opaque plastic) used for EVOO by Rizzo[16] showed rapid degradation of chlorophylls measured at 670 nm under the light of 1 and 4 fluorescent lamps.

Vekiari[17] concluded that storing EVOO in PVC bottles, because of the light and the even low oxygen permeability, should not be suggested as the most appropriate means for maintaining the quality of EVOO.

Pristouri[6] showed that the oxygen transmission rate (OTR) is negligible for glass packaging. For the PET and PP packaging materials, however, the OTRs obtained were 0.9 and 15.6 mL/package per day, respectively, and, in this case, excluding the light effect, the difference between OTRs of packaging materials has a significant effect on the shelf life olive oil.

Piergiovanni[15] informed that metal and glass are the only packaging materials that provide a virtually total barrier to moisture and gases. The word “virtually” is used because such containers require a closure that includes other materials, such as polymeric compounds in cans and in closures, whereby O_2 can easily permeate and promote oxidation of the product. Therefore, it is important to ensure the closure integrity of these products.

It is important to stress that the sources of oxygen, besides packaging wall permeability or closure integrity alteration, are the amount of oxygen dissolved in the oil when bottled and the oxygen in the bottle headspace.[3] To improve better shelf life characteristics, in some products, the amount of oxygen in the headspace can be replaced with nitrogen gas during the filling process and can modify its atmosphere. This is a way to ensure less availability of oxygen and slow down the rate of oxidation.

Glass is widely used for bottling EVOO in Brazil, which is an inert and impermeable material that preserves the product's characteristics during its shelf life. A wide range of olive oil brands are commercialized in Brazil and can be found in glass bottles with different shapes, very attractive designs, coloured or decorated to provide premium positioning for the product and help manufacturers differentiate their products on retail shelf space.[18]

Transparent PET bottles are considered an effective barrier to wavelengths of light shorter than a range of 300 to 340 nm[3,19] but not above this wavelength range, and this can lead to the photo-oxidation of olive oil and reduce its shelf life.

Absorption of light by packaging material (glass or PET) will depend on the colour type itself, colour intensity, and wall thickness due to the fact that a thicker material will absorb more light because of its higher optical density.[20]

Coloured glass can be obtained by adding small quantities of different oxides such as chromium (for green), cobalt (for blue), and others. Amber glass is the most common coloured glass, and it is produced by adding iron, sulphur, and carbon together in a reducing combustion atmosphere melt. Amber glass absorbs nearly all radiation consisting of wavelengths shorter than 450 nm offering excellent protection from ultraviolet radiation (critical for some products sensitive to light).[21] Green glass bottles protect relatively the product from light transmission at a wavelength of approximately 450 nm and above 650 nm.[21]

Considering shelf life of vegetable oils in different packages, Piergiovanni[15] concluded that oil stability can be enhanced by selecting suitable packaging, but little information is available about the characteristics of the packaging material in the UV and visible range.

Therefore, the main aims of this paper are to assess the light barrier in coloured and decorated (label or ceramic decoration) glass bottles suitable for EVOO found on the Brazilian market and evaluate the quality of the packaging used for these products. Oxygen permeability and integrity of the closure system, headspace gas concentration, and the main quality parameter of the EVOO were also evaluated for some glass bottles.

2 | MATERIALS AND METHODS

2.1 | Samples

Twelve different brands of EVOO in glass bottles with a 0.5-L capacity available on the Brazilian market were analysed in this study, according to the description in Table 1. Three samples of glass bottles for each brand made of transparent/clear glass (samples A and B), samples in coloured glass with different shades of colour intensities bottles (samples C to H) and in coloured glass with white and black shrink labels (samples I and J), and samples with external decoration (samples K and L) were analysed.

All samples commercialized in Brazil were originally from different countries such as Portugal (84%), Spain (8%), and Chile (8%) and were acquired from supermarkets in Campinas, São Paulo state, Brazil. All of the selected products had an expiry data within the period of this study, and the shelf life usually established for these products was approximately from 18 months to a maximum of 36 months.

All analysed samples included on its label the information about the type of olive oil, the producers, and country of origin, according to the requirements established in Brazilian Legislation n°1/12.[8] The maximum acidity (%) stated on the label has been informed in Table 1, but no information about the PV or absorption coefficient was provided on the label as specified in the Legislation.[8]

Bottles showed the body diameter ranging from 56 to 71 mm and total height ranging from 198 to 280 mm that were according to the national standardization.[23] Some glass bottles had a special shape to

TABLE 1 Glass bottles used for extra virgin olive oil (EVOO)

Sample	Colour/Decoration	Country/Origin	Maximum Acidity—Stated on the Label (%)
A	Clear/transparent glass	Spain	0.4
B	Clear/transparent glass	Portugal	0.8
C	Coloured glass	Portugal	0.5
D	Coloured glass	Portugal	0.5
E	Coloured glass	Portugal	0.5
F	Coloured glass	Portugal	0.4
G	Coloured glass	Portugal	0.3
H	Coloured glass	Portugal	0.3
I	Coloured glass with white full-body (top to bottom form) PET shrink sleeve label[a]	Portugal	0.2
J	Coloured glass with black full-body (top to bottom form) PET shrink sleeve label[a]	Portugal	0.3
K	Coloured glass bottle covered with a total black ceramic decoration[b]	Chile	0.2
L	Coloured and frosted /etched glass bottle with applied ceramic label[c]	Portugal	0.1

[a]*A shrink sleeve is a full body PVC or PETG pre-printed sleeve that when applied over glass bottles shrinks with heat or steam to fit the body contour of the bottle.*[22]

[b]*Ceramic decoration and applied ceramic labelling is a process where ceramic inks are printed directly onto the bottle and then heated to adhere the ink into the bottle, creating a permanent decoration.*[22]

[c]*Acid etching creates an eye-catching frosted appealing look.*[22]

make this packaging easier to hold while using them. Aluminium Roll-on Pilfer-proof (diameter 31 mm) caps were used in all the analysed glass bottles as a closure system with a tamper evident band.

Light transmission in a spectrum range from 200 and 800 nm of wavelength, which includes the ultraviolet (<400 nm) and the visible light (400-700 nm), was evaluated for all EVOO bottles after selecting a representative flat piece of the sidewall for analysis.

The National Standard ABNT NBR 14910[23] also specifies a classification in transparent or coloured (amber or green) glass bottles according to a light transmission reference (*Tr*), determined at 550 nm of wavelength (λ) with posterior correction of the value for a specific thickness (3.5 mm for transparent glass or 3.0 mm for amber/green glass). Light transmission was calculated by the equation below:

$$\log\left(\frac{\mathrm{Tr}}{0,92}\right) = \left(\frac{\mathrm{er}}{\mathrm{e1}}\right)\log\left(\frac{\mathrm{T1}}{0,92}\right)$$

where:

"*Tr*" is the light transmission reference, in %.

"*er*" is a specific thickness, in mm.

"e_1" is the real thickness of the sample, in mm (measured at the point of light incidence).

"T_1" is the light transmission obtained for the analysed sample (applied in the equation in absolute value, not in percentage).

According to the value obtained for the transmission reference (Tr) at 550 nm, samples can be classified as clear/transparent glass if *Tr* is above or equal to 85%, amber glass if *Tr* is between 20% and 35% and green glass if *Tr* is between 70% and 80%.[23]

All light transmission results were obtained using a spectrophotometer UV/Vis (ultraviolet/visible)—Analytik Jena (Thuringia, Germany), model Specord 210. The glass wall thickness was measured using a Mitutoyo calliper (Takatsu-ku, Japan), with a resolution of 0.01 mm.

Glass composition and ceramic decoration/inks were identified and semi-quantified relatively by a SEM/EDX—Scanning Electron Microscopy with energy dispersive X-ray spectroscopy, model DSM 940a, using a system Link ISIS—Suite Revision 3.35 (Oxford, UK). The semi-quantification of the elements was classified as majority (>10%), minority (between 10% and 1%), and trace (<1%).[24] Microanalysis was obtained using the same parameters of X-ray (work distance 25 mm and voltage 20 kV) with a beryllium window in which the chemical elements with an atomic number (Z) equal or higher than 11 could be detected.

2.2 | Closure integrity test, oxygen transmission rate (OTR), and headspace gas analysis

Three units of glass bottles for each brand, sample A, B, C, E, and G, were analysed for the closure integrity, OTR, and headspace gas analysis.

The quality and airtightness of the closure system were evaluated by helium leak testing with a mass spectrometer detector probe adapted from the ASTM E499/E499M[25] methodology. An equipment produced by BOC *Edwards* (Crawley, UK), model *Spectron* 5000, stated with a detection limit of 1×10^{-10} mbar.L.s^{-1} was used. The unit mbar.L describes the amount of gas regardless of the pressure, and 1 mbar.L.s^{-1} corresponds to a flow of 1 mL per second at 1 bar, which is equivalent to 0.1 Pa.m^3 s^{-1}.[26] Samples with the closure system (as acquired) were filled manually with industrial helium gas in a hood, after piercing the bottom, removing the product, and re-sealing the hole with a silicon septum, which was checked for sufficient tightness.

The OTR was determined using the OX-TRAN, model 2/60, Oxygen Permeability Tester Mocon (Minneapolis, USA) at a temperature of 22°C.[27] The results were expressed as mL/(package day).

The headspace gas concentration, especially oxygen (O_2) and nitrogen (N_2), were identified and quantified by using a gas chromatograph Agilent, model 78990 (Santa Clara, USA) with a thermal conductivity detector at 150°C, 13X molecular sieve column Porapak C at

40°C and injector at 70°C. Chemstation/Agilent software, version 03.01B, based on a standard curve made with a mixture of calibration gases, Praxair (Danbury, USA) was used for gas identification and quantification. The results were expressed in percentage of gas volume (% *v*/*v*) at 22°C and 707 mmHg.

2.3 | Analytical measurements to evaluate the quality of the EVOO

The chemical parameters of the EVOO were determined in triplicate for each brand, following the analytical methods described in The American Oil Chemists' Society—AOCS Official Methods. Ethanol and sodium hydroxide from Merck (Darmstadt, Germany) and phenolphthalein were used as indicators from Sigma-Aldrich (St. Louis, USA) for the maximum acidity (%), expressed as a percentage of oleic acid.[28,29] The peroxide value (PV) was determined by using sodium thiosulfate from Merck, and its results were given in meq O_2/kg oil (milliequivalents of active oxygen per kilogram of oil).[30] Cyclohexane solvent from Merck was used for absorption coefficient (K_{232}, K_{270}) determination using a UV-Vis spectrophotometer from Varian (California, USA).[31]

3 | RESULTS

3.1 | Light barrier properties of the glass bottles, glass colour classification, and glass composition

Figure 1 shows the light transmittance for 2 transparent/clear glass bottles in a wall thickness approximately 2.6 to 2.7 mm. Transparent/clear glass provides a total light barrier in the ultraviolet range below 320 nm in which light transmission does not occur, similar to PET bottles, according to Cecchi et al[10] and Coltro et al.[19]

Wavelengths of light above 320 nm cannot be absorbed by transparent glass packaging, and, when exposed to light, the whole range of visible light of the wavelengths between 320 and 700 nm can reach the product by a maximum percentage of 90%. Light transmittance even in transparent glass will never be higher than 92% because each surface presents a reflection in the order of 4%.[20]

Light transmission reference (Tr) at 550 nm obtained for sample A and B was 87% and 86%, ie, higher than 85% and therefore attends the specification as transparent glass according to the National Standard.[23] Rizzo[16] also demonstrated that clear plastic bottle showed a light transmittance of 80% to 90% from 400 to 700 nm.

According to the maximum light absorption by natural photosensitizers, ie, chlorophyll in EVOO (between 400 and 500 nm and between 600 and 700 nm),[14] there is no protection for the product in transparent glass in these wavelength ranges.

Transparent glass packaging is widely used in food marketing because the conditioned product can be seen. However, no protection to the light on the wavelength of visible light is provided by transparent packaging, especially if it is marketed under light storage conditions. Clear glass allows the direct action of light on the olive oil, and this could promote oxidative rancidity because of its sensibility to photo-oxidation.[17]

Figure 2 shows light transmission spectra for coloured glass bottles (samples C, D, E, F, G, and H) with different shades in colour (lighter or darker). The specular light transmission values for some wavelengths and the average thickness and the reference transmittance at 550 nm for each sample are shown in Table 2.

The spectrum presented absence of light transmittance below 400 nm for all samples regardless of the glass colour shade or intensity and therefore total light barrier at the ultraviolet radiation. Above 400 nm, a higher light transmittance was in the wavelength ranged from 550 to 600 nm with a maximum of 38.8% for sample D, followed by sample C, E, F, G, and H. This is exactly the sequence of glass colour shade perceived as lighter (sample D) to darker (sample H) when seen with the naked eye. This behaviour is related to the least colour intensity and the smallest wall thickness values observed in sample D when compared with the other samples.

Light transmission reference (Tr) at 550 nm obtained for samples C to G showed results between 20% and 33%, and according to ABNT NBR 14910,[23] they can be classified as amber glass (Tr for amber glass is between 20% and 35%). There is a visual perception at retail stores that, especially samples C, D, E, and F, are green glass bottles, but according to the National Standard they are classified as amber glass bottles. Samples G and H are similar in that there is a visual perception of black coloured bottles in retails stores. However, sample G is still classified as amber glass due to its 20% of light transmission reference at 550 nm (Tr). Only sample H showed 10% of transmission reference (Tr) at 550 nm, due to its intense dark colour, and there is no classification in the National Standard.

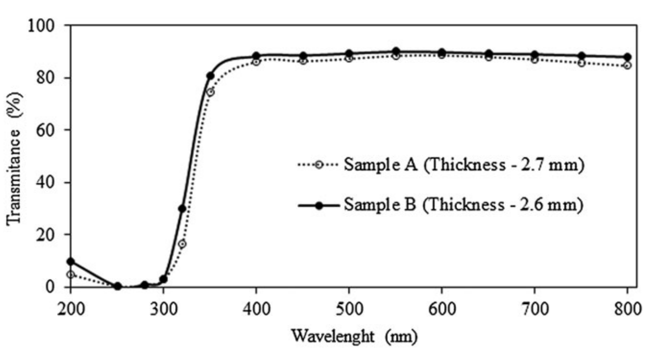

FIGURE 1 Spectrum of light transmittance (%) of transparent/clear glass bottles

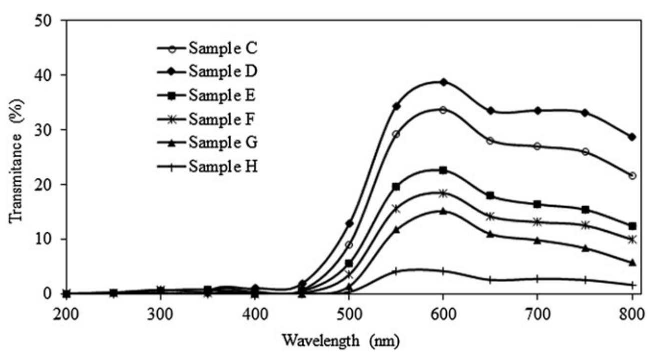

FIGURE 2 Spectrum of light transmittance (%) of coloured glass bottles (samples C, D, E, F, G, and H)

TABLE 2 Specular light transmission (%) for coloured glass bottles with different shades in colour, average thickness, and reference transmittance at 550 nm

	Sample					
Wavelengths, nm	C	D	E	F	G	H
200	0.0	0.0	0.0	0.0	0.0	0.0
300	0.0	0.0	0.0	0.0	0.0	0.0
400	0.4	1.1	0.3	0.1	0.1	0.0
450	0.7	1.9	0.6	0.2	0.1	0.0
500	9.1	12.9	5.7	3.6	1.4	0.4
550	29.2	34.4	19.7	15.7	11.8	4.2
600	33.7	38.8	22.6	18.5	15.2	4.2
650	28.1	33.5	18.0	14.2	11.0	2.6
700	27.0	33.6	16.4	13.2	9.9	2.8
800	21.7	28.7	12.5	10.0	5.8	1.7
Average thickness (mm)	3.3	2.9	3.2	3.7	4.1	4.2
Reference transmittance at 550 nm	32	33	22	22	20	10

The characterization of the glass composition by SEM/EDX showed the chemical elements Si—silicon (67%-71%), Na—sodium (13%-15%), and Ca—calcium (11%-13%) that was classified as majority. Minor additives classified as minority such as Mg—magnesium, Al—aluminum, and K—potassium with relative percentage between 1% and 3% were also determined. Therefore, all these samples (A to H) of glass bottles were classified as soda-lime glass. A percentage of the Fe—iron chemical element was detected around 0.4% for sample E, and 0.7% and 0.6% for samples G and H, generally used as a pigment to obtain the amber colour.

At the maximum light absorption by chlorophyll, coloured glass bottles can protect the product from light incidence from a wavelength 400 to 500 nm range from 0.0% to 12.9% and from a wavelength 600 to 700 nm range from 2.6% to 38.8%. Light protection is more significant for glass in dark colours (sample H).

Rizzo[16] demonstrated that the best light barrier property was for the white PET bottle (opaque plastic), but it still showed a light transmittance around 20% to 30% in the chlorophyll light absorption range (400-500 nm and 600-700 nm). Green, orange, and blue PET bottles showed a light transmittance around 50% to 80% in the same wavelength range.[16] Although the colour may appear darker, it does not always have a sufficient light barrier to protect the product at a specific wavelength.

Figures 3 and 4 show light transmission spectra for coloured glass bottles with white and black sleeve (samples I and J) and decorated glass bottle (samples K and L), respectively.

Coloured glass with black label (sample J) and coloured glass with a black ceramic decoration (sample K) showing total light barrier and no light transmittance were observed from 200 to 800 nm wavelength range.

Coloured glass with white label (sample I) showed a small light transmittance after 450 nm with a maximum of 8% around 550 to 600 nm wavelength (Figure 3). However, coloured glass with frosted effect decoration (sample L) did not provide the light protection necessary for the product and a light transmittance starting from around 320 nm wavelength to a maximum of 25% between 550 and 600 nm (Figure 4).

Shrink labels and ceramic decoration provide an additional physical light barrier to coloured glass, while the frosted effect decoration causes only a light scattering and a no effective protection for the product against light.

At the maximum light absorption by chlorophyll, sample I and L can protect the product from light incidence from a wavelength 400 to 500 nm range in 4% and 9% and from wavelength 600 to 700 nm range approximately 7% and 25%, respectively.

Despite its label or decoration, samples I, J, and L are also classified as amber glass according to the National Standard due to its light

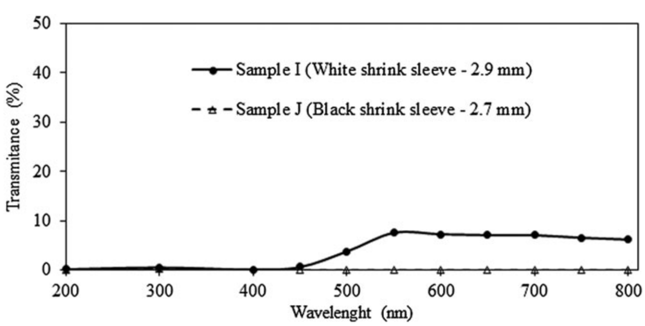

FIGURE 3 Spectrum of light transmittance (%) of coloured glass with white (sample I) and black (sample J) shrink sleeve

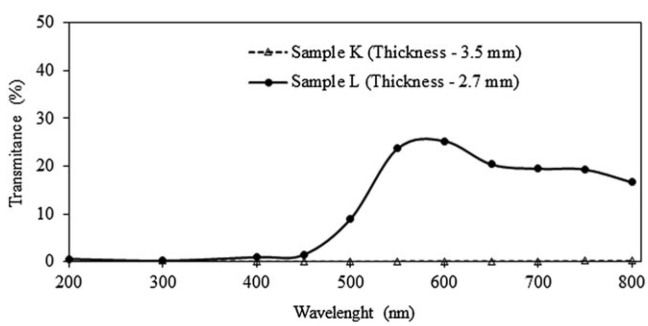

FIGURE 4 Spectrum of light transmittance (%) of coloured glass with ceramic/frosted external decoration (samples K and L)

transmission reference at 550 nm (Tr) was 22% and 26%. Sample K may not be classified due to the external ceramic decoration.

These glass bottles (samples I to K) also showed a chemical composition similar to amber glass and were classified as soda-lime glass. Other majority chemical elements such as Si—silicon (90%) and S—sulphur (8%) for sample K in the external decoration and Ti—titanium (12%) and Z—zinc (19%) for sample L were identified by SEM/EDX.

Caponio[12] ascribed that EVOO in clear glass bottles under light conditions showed a decrease in PV due to evolution from primary to secondary oxidation process. Vekiari[17] affirmed that glass bottles, if stored in a dark condition, maintained a better quality of EVOO and provided higher protection from oxidation due to the fact that glass acts as a barrier to oxygen and avoids losing certain components that deteriorate the EVOO under its presence. They also concluded that the packaging material should ensure protection from light in order to maintain the quality of the olive oil.

Therefore, a better protection and maintenance of the EVOO quality can be obtained in glass bottles with higher light barrier properties when associated with an efficient closure system.

TABLE 4 Acidity[a], peroxide value,[a] and absorption coefficients[a] at 232 and 270 nm

Samples	Acidity, %	Peroxide Value—PV, meq O_2/kg oil	Absorption Coefficients K_{232}	K_{270}
A	0.34 ± 0.01	8.71 ± 0.15	2.00 ± 0.02	0.22 ± 0.02
B	0.59 ± 0.01	11.15 ± 0.26	2.20 ± 0.01	0.18 ± 0.01
C	0.75 ± 0.01	8.84 ± 0.19	2.02 ± 0.03	0.18 ± 0.01
D	0.74 ± 0.01	9.49 ± 0.57	2.09 ± 0.07	0.16 ± 0.01
E	0.54 ± 0.01	8.25 ± 0.14	2.09 ± 0.02	0.21 ± 0.01
F	0.71 ± 0.02	6.54 ± 0.16	2.12 ± 0.01	0.14 ± 0.01
G	0.82 ± 0.01	10.52 ± 0.32	2.16 ± 0.05	0.19 ± 0.01
H	0.68 ± 0.01	7.55 ± 0.14	2.18 ± 0.08	0.23 ± 0.01
I	0.36 ± 0.02	7.85 ± 0.30	2.06 ± 0.02	0.20 ± 0.01
J	0.42 ± 0.01	5.84 ± 0.10	2.12 ± 0.06	0.16 ± 0.01
K	0.36 ± 0.02	6.14 ± 0.16	1.98 ± 0.09	0.15 ± 0.01
L	0.40 ± 0.01	7.96 ± 0.16	2.08 ± 0.01	0.24 ± 0.02
Required limits[b]	≤0.80	≤20	≤2.50	≤0.22

[a]*Standard deviation of 3 replicates on the same sample.*

[b]*Required limits at legislation.*

3.2 | Closure integrity, oxygen transmission rate (OTR), and headspace gas analysis

The results of helium leakage rates, OTR, and headspace gas analysis are shown in Table 3.

The aluminum Roll-on Pilfer-proof caps used for the EVOO glass bottles showed helium leakage rates between 1.3×10^{-6} mbar.L.s^{-1} and 1.6×10^{-10} mbar.L.s^{-1}.

Helium leakage rates of 3.6×10^{-6} mbar.L.s^{-1} and 8.2×10^{-6} mbar.L.s^{-1} were obtained by Kossinna[27] when analysed glass bottles with PP—a pilfer proof cap 28 mm neck finish, closed in a fast speed production line and closed manually, respectively. These results indicate a good integrity of the closure system used for the analysed EVOO glass bottles.

The OTR results for the analysed glass bottles with its commercialized closure system were between 0.017 and 0.038 mL / (package day) at a temperature of 22°C. Pristouri[6] commented that the OTR of glass is negligible when compared with PET or PP bottles and these results indicate that the closure system used for the analysed EVOO glass container has low oxygen permeability, because glass is impermeable and has a good barrier to oxygen.

The headspace gas analysis shows average percentage of oxygen (O_2) between 1.2% and 6.6% and nitrogen (N_2) between 89.2% and 94.8% in the analysed glass container.

The average composition of air is approximately 21% of oxygen, 78% of nitrogen, and 1% of other gases, and the previous results show that the analysed EVOO glass containers had a modified atmosphere. The variability of some results can be associated with an eventual inefficient process as the oxygen permeation through the closure system was very small. Guil-Gerrero[13] affirmed that EVOO stored under nitrogen atmosphere could be packaged in glass bottles without appreciable quality changes.

3.3 | EVOO quality—acidity, PV, and absorption coefficients K_{232} and K_{270}

The analytical measurements obtained by the EVOO are reported in Table 4. All the products showed results below the requirements established by the legislation[8,9] for acidity (≤0.80%) and PV

TABLE 3 Helium leakage rate, OTR, and headspace gas analysis results

Test		Values[a]	Sample A	B	C	D	G
Helium leakage rate (mbar.L.s^{-1})[b]		Minimum leakage flow	1.2×10^{-8}	1.6×10^{-10}	2.0×10^{-8}	9.8×10^{-9}	1.6×10^{-10}
		Maximum leakage flow	3.5×10^{-8}	2.0×10^{-10}	1.0×10^{-6}	1.3×10^{-6}	1.2×10^{-7}
OTR (mL / (package day) at 22°C)		Average	0.038	0.028	0.017	0.027	0.033
		Standard deviation	± 0.002	± 0.030	± 0.012	± 0.014	± 0.002
Headspace gas analysis (%)	O_2	Average	1.9	1.2	2.1	2.0	6.6
		Standard deviation	± 1.2	± 0.4	± 0.3	± 0.7	± 2.3
	N_2	Average	91.9	92.2	94.8	94.4	89.2
		Standard deviation	± 1.6	± 0.6	± 2.6	± 3.2	± 2.4

[a]*Results of 3 glass bottles for each brand.*

[b]*1 mbar.L.s^{-1} = 0.1 Pa.m^3.s^{-1}.*

(≤20 meq O_2/kg oil), except for the product in sample G that presented an acidity mean value of 0.82% and slightly above the limit.

Regarding acidity values for EVOO, Caponio[12] concluded that the acidity remained virtually constant during storage, and no significances were observed between the 2 storage conditions (under diffuse light or in the dark). Vekiari[17] investigated the effect of the extraction process in the quality of the EVOO and observed significant changes in some parameters of the product including acidity, PV, UV absorbance, and others.

The acidity results obtained for EVOO in sample G probably can be associated to production process of the product, because glass bottle, classified as amber colour, would still protect the product at approximately 85% at 600 to 700 nm even if storage under at light. Sample G was also manufactured in a period before the other analysed samples and had the highest shelf life of 36 months.

The values of absorption coefficients were between 1.98 and 2.20 for K_{232}, within the limits permitted by legislation, and between 0.14 and 0.24 for K_{270}, ie, 2 samples (H and L) with levels of absorbance at 270 nm were above the limit of 0.22. According to Méndez[5] and Guil-Gerrero,[13] the K_{270} alteration can indicate that oil oxidation has begun, and it results from an initial degradation process of hydroperoxides, which could be due to some factors affecting storage conditions or inadequate EVOO processing.

Because the PV attended the required limit by legislation and all the analysed samples had an expiry data within the period of this study, these small variations in the EVOO parameters can probably be associated with the quality of raw material used to obtain the product and/or its manufacture process.

When the acidity results determined from the product were compared with the maximum acidity described on the glass bottle's label (Table 1), it could be observed that only samples A and B maintained the correct information about acidity on the label as the results obtained were below the stated information. The other samples showed acidity values above the value declared on the label, although the EVOO reached the limit specified for acidity by the legislation, exception for sample G, as informed previously.

4 | CONCLUSION

Transparent or clear glass has no efficient light barrier for EVOO products and can lead to photo-oxidation of the product when stored under commercial conditions.

Packaging material should ensure protection from light in order to slow down the oxidation process and maintain the quality of the olive oil. Moreover, coloured glass bottles exhibit a good light barrier property. An increase in light barrier property was observed for the darkest coloured packaging and the variability in the colour of glass depends on the pigment quantity and the wall thickness. In general, coloured glass bottles for EVOO commercialized in Brazil are classified as amber, although the glass looks greenish with different shades.

Coloured glass bottles with black shrink sleeves and glass bottles with ceramic external decoration showed a total light barrier from a wavelength 200 to 800 nm range. Decorated glass bottles may improve glass container properties regarding quality retention of olive oil due to its better effective light barrier property.

Using white shrink sleeves in coloured glass bottles showed relative light barrier properties and relative protection of the EVOO from light.

The analysed glass bottles for EVOO commercialized in Brazil demonstrated a low oxygen permeability, a good closure integrity, and had a modified atmosphere to avoid some reaction of deterioration in the product by the oxygen. In general, the analysed products showed results for quality parameters that met the requirements set out in Brazilian Regulations.

It is important to emphasize, however, that packaging should be selected according to the needs of product protection against undesirable changes. Moreover, it is extremely important to develop studies addressing the product's shelf life in the selected packaging aiming at ensuring the effectiveness of conservation and the quality of the conditioned product.

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ORCID

S.B.M. Jaime http://orcid.org/0000-0002-7558-4966

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