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# Physicochemical and microbiological assessment of commercial dehydrated black olives

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ABSTRACT

Among the many different types of table olives, the production of dehydrated black olives is rather low worldwide, although there is an increasing interest in them from consumers. The physicochemical and microbiological characterization of 20 samples of these olives obtained from different countries and processing methods was studied. The average pH and water activity ($a_w$) values were 4.7 units and 0.85 respectively, although a great variability was found among samples due to the cultivar and the elaboration method used, including oven-dried olives and dry-salted olives obtained from harvested fruit along with oven-dried fruit from fermented black olives. Moreover, the NaCl concentration in many samples was higher than the unique legal requirement for these olives by international standards (>8–10% in the olive juice), which probably implies a reduction of this mineral in the future as demanded by consumers. More precisely, this product must be valorized based on its 40% content of oil rich in monounsaturated fatty acids, and more than 2500 mg/kg of phenolic compounds and 3500 mg/kg of triterpenic acids which make these olives a very attractive product from a nutritional point of view.

## 1. Introduction

While the worldwide annual production of olive oil and table olives is rather similar at around three million tons each, the quantity of research focused on olive oil has traditionally been much greater in comparison to studies on table olives. Most commercial table olives worldwide are elaborated following the (i) green olives or Spanish-style, (ii) black olives or California-style, and (iii) natural black olives or Greek style. However, there is an increasing consumer interest in dehydrated foods, particularly dehydrated olives for snacks, bread, mix of dehydrated vegetables and many other uses. Traditionally, most of the dehydrated black olives have been elaborated from black overripe fruit that are placed in tanks with coarse salt (NaCl) for one to two months (Panagou, 2006). During this period, the osmotic dehydration leads to water loss, enrichment in NaCl and rupture of tissues. Consequently, bitterness is reduced because oleuropein contacts with the polyphenoloxidase enzyme (Ramírez et al., 2013).

Hot air drying is also applied in some countries to produce dehydrated black olives, as for example the "Ferrandina" method in Italy that includes blanching, salting and oven-drying of mature olives (Cardoso et al., 2009; Marsilio et al., 2000). Moreover, oven dehydration is carried out on fermented black olives in Peru (De Florio-Ramírez & Lanchipa-Bergamini, 2008) and open-air dehydration of harvested black olives is performed in some regions of Algeria (Boukhiar et al., 2017).

Both dehydration methods, salting and heating, are static and discontinuous, although the dry-heating process allows the elaboration of olives during the whole year from fermented fruit. Obviously, it must be considered the cost of the power to dehydrate the olives but the environmental impact of this process is lower than that generated by the traditional dry-salting.

Dehydrated black olives are packed and marketed in glass containers or plastic bags without brine so that their safety during storage is assured due to their low water activity/high NaCl content. In fact, international table olive standards only regulate the level of NaCl in the olive juice at a minimum of 10% (IOC, 2004) or 8% (Codex Alimentarius, 2013). It must be taken into account that the pH of these olives is currently higher than 4.5 units but *Clostridium botulinum* does not grow at NaCl content higher than 10% (Kim & Foegeding, 1993; Wareing & Fernandes, 2007).

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However, it is very difficult to analyze the concentration of NaCl in the juice of dehydrated olives because of the low amount of this liquid that cannot be obtained even using centrifugation, so NaCl is currently measured in a mixture of olive pulp and distilled water, but the content of NaCl in the olive juice is not referred to as required by standards (Değirmencioğlu et al., 2014; Panagou et al., 2006).

Physicochemical, microbiological and sensorial characterization of commercial table olives of the three main elaboration types (green, black and natural black olives) is available in the literature for Spanish (López-López et al., 2004), Greek (Panagou et al., 2006), Portuguese (Pereira et al., 2008; Pires-Cabral et al., 2018), Italian (Franzetti et al., 2011) and Turkish olives (Yilmaz & Aydeniz, 2012). By contrast, there is very little information with regard to the quality profile of dehydrated black olives (Panagou et al., 2006) that could be of great interest for processors and consumers along with table olive standards regulators and it could contribute to improve labelling and food traceability. In addition, table olives are a good source of bioactive substances including phenolic compounds (Boskou et al., 2006; Romero et al., 2004) and triterpenic acids (Romero et al., 2010; Alexandraki et al., 2014) with important biological properties (Piscopo et al., 2014; Rufino-Palomares et al., 2022; Yu et al., 2021). Again, only scarce data are available on the phenolic content of dehydrated black olives (Mantzouridou & Tsimidou, 2011; Ramírez et al., 2013; Zoidou et al., 2010) and no information can be found on the concentration of triterpenic acids in these olives.

The objectives of this work were (i) to characterize the physico-chemical and microbiological profile of commercial dehydrated black olives purchased worldwide, particularly the compliance of this product with international standards, and (ii) to valorize this food based on its content of bioactive substances.

## 2. Materials and methods

### 2.1. Samples

Duplicate of twenty samples of dehydrated black olives were purchased from supermarkets and online markets worldwide (Table 1). All of them were packed without cover brine in jars or pouches, and the best before consumption advice ranged between 1 and 3 years. It must be noted that olives were elaborated in the main producing countries of dehydrated table olives (Turkey, Greece, Morocco, Spain, Italy and Peru).

### 2.2. pH and moisture

Twenty previously pitted olives were chopped and homogenized in an Ultra-Turrax homogenizer, T25, IKA-Laborthechnik (Staufen, Germany). The pH value of the paste was determined with a Crison Basis 20 pH-meter (Barcelona, Spain).

Olive moisture was measured by oven drying three portions of the homogenized paste (5 g) at 105 °C up to constant weight.

### 2.3. NaCl content

Thirty grams of chopped olive pulp was mixed with 30 mL of distilled water and homogenized in an Ultra Turrax homogenizer. The homogenate was squeezed through cheesecloth and the concentration of NaCl in the aqueous phase was analyzed by titration with a 0.86 N silver nitrate solution, using a potassium chromate solution as indicator. The value obtained by titration was multiplied by 2 and it was expressed as the percentage of NaCl per 100 g of olive flesh (g NaCl/100 g pulp).

Supposing that all the NaCl was concentrated in the aqueous phase of the olive (juice), the NaCl content of the olive juice was calculated as follows:

$S_j = S_m \times (100 + M/M)$

**Table 1**
Commercial samples of dry black olives and sorbic acid content.

| Sample | Country (comerzialization/ origin) | Type of packaging | Package size (g) | Additive on label | Sorbic acid (mg/kg) |
|---|---|---|---|---|---|
| 1 | Spain/Spain | Jar | 210 | None | Not detected |
| 2 | Spain/Spain | Jar | 250 | None | Not detected |
| 3 | Spain/Spain | Pouch | 500 | None | Not detected |
| 4 | UK/Morocco | Jar | 110 | Potassium Sorbate | 1021 (55)[c] |
| 5 | Greece/Greece | Pouch | 250 | None | Not detected |
| 6 | Spain/Spain | Jar | 190 | Lactic and ascorbic acids | Not detected |
| 7 | Turkey/Turkey | Pouch | 130 | None | Not detected |
| 8 | Australia/Italy | Jar | 350 | Lactic acid | 33 (2) |
| 9 | Australia/Italy | Jar | 325 | Citric acid | 372 (6) |
| 10 | Peru/Peru | Pouch | 227 | None | Not detected |
| 11 | Peru/Peru | Pouch[a] | 227 | None | Not detected |
| 12 | Turkey/Turkey | Jar | 120 | None | Not detected |
| 13 | UK/Morocco | Jar | 250 | None | Not detected |
| 14 | Italy/Italy | Pouch[b] | 250 | None | Not detected |
| 15 | Germany/? | Jar | 190 | None | Not detected |
| 16 | Germany/? | Jar | 230 | None | Not detected |
| 17 | Germany/ Morocco | Pouch | 200 | None | 18 (1) |
| 18 | Germany/? | Pouch | 250 | None | Not detected |
| 19 | Germany/Turkey | Rigid pouch | 400 | Lactic acid and $CaCl_2$ | Not detected |
| 20 | Greece/Greece | Rigid pouch | 500 | None | Not detected |

[a] Pitted olives.
[b] Storage under refrigeration.
[c] Standard deviation of duplicates is shown between parenthesis.

$S_j$ = NaCl (g/100 mL) in the olive juice

$S_m$ = NaCl in the mixture olive/distilled water (1:1)

M = moisture

### 2.4. Water activity ($a_w$)

Water activity of the olives was analyzed with an AquaLab Pre apparatus (Meter Group Inc., WA, USA). Two or three pieces of olive pulp were placed in a disposable sample cup and $a_w$ was measured in triplicate.

### 2.5. Oil content and oxidation degree

The oil content in the olive pulp was determined by NMR using a minispec mq 100 Bruker apparatus (MA, USA) (García-Sánchez et al., 2005).

The peroxide value and specific ultraviolet (UV) absorbance ($K_{232}$ and $K_{270}$) were measured following the analytical methods described in EC Regulation 2568/91.

### 2.6. Olive quality

The color of olives was measured using a HunterLab ColorFlex EZ spectrophotometer, equipped with computer software to calculate the CIE $L^*$ (lightness), $a^*$ (redness), and $b^*$ (yellowness) parameters. The reflectance at 700 nm was also recorded. Interference by stray light was minimized by covering samples with a box that had a matte black interior. The data from each measurement is the average of 10 olives.

The firmness of fruit was determined using a Kramer Shear Cell of one blade coupled to a Texture Analyzer TA.TX plus (Stable Microsystems, Godalming, UK). Two halves of each olive were placed facing the blade. The crosshead speed was 200 mm/min. Firmness was the mean of 10 replicate measurements and expressed as Newton/fruit.

### 2.7. Atmosphere in jars and pouches

Concentrations of $O_2$ and $CO_2$ in the inner atmosphere of containers were measured with a Gaspace Advance Headspace/MAP Analyzer (Illinois Instruments, Inc. Jojnsburg, IL, USA) by introducing a hypodermic needle into plastic pouches or a steel piercing head into the metallic closure of glass jars. This analysis was carried out on the arrival of the samples at the laboratory.

### 2.8. Sorbic acid

It was determined following the method described elsewhere (Brenes et al., 2004). Briefly, 1 g of triturated olive pulp was mixed with 25 mL of 0.2 M NaOH, the mixture being agitated in a vortex and centrifuged. After acidification and filtration, 20 μL was injected into the chromatograph. A HPLC Waters 2690 Alliance apparatus (Waters Inc) was used with a pump, column heater, autosampler and a Waters 996 photodiode array detector. A 25 cm × 4.5 mm i. d., 5 μm Sherisorb ODS-2 column (Wasters Inc.) was employed. Separation was achieved by gradient elution using an initial composition of 90% water with of 0.005 ammonium acetate buffered at pH 4.2 with glacial acetic acid (A), and 10% methanol (B). A flow rate of 1 mL/min and a temperature of 35 °C were used. Chromatograms were recorded at 260 nm.

### 2.9. Bioactive substances

Phenolic compounds were extracted from the olive pulp with dimethyl sulfoxide (DMSO) according to García et al. (2018). Briefly, small pieces of pulp (<0.5 g) were cut from 10 olives up to a total of 10 g that were introduced into a solution of 30 mL of DMSO. The mixture was crushed with an Ultra-Turrax homogenizer and, after 30 min of resting contact, it was centrifuged at 6000 g for 5 min, and 0.25 mL of the supernatant was diluted with 0.5 mL of DMSO plus 0.25 mL of 0.2 mM syringic acid (internal standard). The mixture was filtered through a 0.22 μm pore size nylon filter, and an aliquot (20 μL) was injected into the same HPLC chromatograph used for sorbic acid analysis.

Triterpenic acids were analyzed following the procedure described by García et al. (2018). One gram of dried olive was mixed in a 10 mL centrifuge tube with 4 mL of methanol/ethanol (1:1, v/v) and vortexed for 1 min, centrifuged at 6000 g for 5 min, and the solvent was separated from the solid phase. This step was repeated six times, and the pooled solvent extract was vacuum evaporated. The residue was dissolved in 4 mL of methanol, which was filtered through a 0.22 μm pore size nylon filter, and an aliquot (20 μL) was injected into the same HPLC chromatograph used for sorbic acid analysis.

### 2.10. Microbiological analyses

The viable and culturable microbial population was achieved by mixing 5–7 olives (15–25 g of fruit) with 50 mL of buffered peptone water (10 g/L peptone, 5 g/L NaCl, 1.5 g/L potassium dihydrogen phosphate, 3.5 g/L disodium hydrogen phosphate, adjusted at pH 7) at 25 °C for 30 min with agitation. One mL or 50 μL of the suspension was plated in PCA agar (Oxoid, Basingstoke, Hampshire, UK), MRS agar (Oxoid) supplemented with 0.2 g/L sodium azide (Sigma-Aldrich, St. Louis, MO, US), VRBD agar (Oxoid) and oxytetracycline-glucose-yeast extract agar (Oxoid) for the enumeration of mesophilic aerobic microorganisms, lactic acid bacteria (LAB), *Enterobacteriaceae*, and yeast and molds, respectively. *Enterobacteriaceae* were incubated at 37 °C for 24 h, mesophilic aerobic bacteria, LAB, and yeasts were set at 32 °C for 48 h (even 5 days when no growth occurred), and the numbers of colony-forming units were counted with a Scan 500 colony counter (Interscience, Saint-Nom-la-Bretèche, France).

## 3. Results and discussion

As reflected in Table 1, all dehydrated black olives were packed without cover brine in both jars and pouches as containers. Pitted olives were only found in sample 11, and seasoned olives in samples 2, 6 and 8. Generally, these dehydrated olives are immersed in vegetable oil before packing as recorded on many of the labels. Moreover, the presence of some additives must also be noted (calcium chloride, potassium sorbate and lactic, ascorbic, and citric acids) in samples 4, 6, 8, 9 and 19, which means that the olives were submerged in a solution with these additives before packaging. However, it is not clear what the objective was for the addition of these additives to dehydrated black olives, except for potassium sorbate. Sorbic acid is a well-known inhibitor of the growth of yeasts and molds, widely employed by the table olive industry (Panagou, 2006). This preservative was only declared in sample 4, where it was found at a concentration slightly higher than the maximum of 1000 mg/kg established by the European Union (EC Regulation 1333/2008). By contrast, it was found in sample 9 and also in low amounts in samples 8 and 17 where it was not declared on labels. Obviously, this additive was intentionally added in sample 9 at a high concentration, and it could be supposed that in samples 8 and 17 it degraded during the storage of the commercial samples and was not detected at the time of analysis. However, it has been reported for green olives that sorbic acid was stable in pasteurized samples and gradually disappeared in unpasteurized samples (Casado et al., 2010) with an increase in the off-flavor *trans*-4-hexenoic acid as a consequence of LAB growth. It must be

**Table 2**
Microbiological analysis of commercial dry black olives (CFU/g).

| Sample | Yeast + mold | Lactic acid bacteria | Mesophilic aerobic bacteria | *Enterobacteriaceae* |
|---|---|---|---|---|
| 1 | BDL[a] | BDL | 3 (1) | BDL |
| 2 | 47 (5)[b] | BDL | 106 (5) | BDL |
| 3 | 25 (2) | BDL | 25 (1) | BDL |
| 4 | BDL | BDL | BDL | BDL |
| 5 | 5384 (210) | BDL | 5384 (550) | BDL |
| 6 | 29 (3) | BDL | 29 (3) | BDL |
| 7 | 9101 (1002) | BDL | 6354 (657) | BDL |
| 8 | BDL | BDL | BDL | BDL |
| 9 | BDL | BDL | BDL | BDL |
| 10 | BDL | BDL | BDL | BDL |
| 11 | BDL | BDL | BDL | BDL |
| 12 | BDL | BDL | BDL | BDL |
| 13 | BDL | BDL | BDL | BDL |
| 14 | 11500 (850) | BDL | 8780 | 2570 |
| 15 | BDL | BDL | BDL | BDL |
| 16 | BDL | BDL | BDL | BDL |
| 17 | BDL | BDL | BDL | BDL |
| 18 | BDL | BDL | BDL | BDL |
| 19 | BDL | BDL | BDL | BDL |
| 20 | 3520 (170) | BDL | 54200 (1560) | BDL |

[a] BDL, below the detection limit that ranged between 2.0 and 3.3 CFU/g.
[b] Standard deviation of duplicates is shown between parenthesis.

remarked that these microorganisms were not detected in any of the samples studied (Table 2) nor was off-flavor found, so this preservative was not added in most of the samples analyzed. In fact, samples could have been pasteurized although this was not indicated on label, so the addition of sorbic acid was unnecessary to stabilize the product.

The average pH of the commercial samples of dehydrated black olives was 4.7 units (Table 3), slightly lower than 5.0–5.2 units reported in some black dry-salted olives (Panagou, 2006; Ramírez et al., 2013) but higher than the minimum limit of 4.5 reported for the growth of *C. botulinum* (Kim & Foegeding, 1993) so additional hurdles must be considered to ensure the microbial stability of the product. It is worth mentioning that for samples 10 and 11 had a very low pH (3.65 and 3.30) due to the lactic acid fermentation process that these olives underwent prior to the oven dehydration stage (information provided by processor) (De Florio-Ramírez & Lanchipa-Bergamini, 2008), thereby this low pH alone could assure the product stability. Likewise, the highest pH was detected in sample 14 (6.10 units) that corresponded to Italian olives dehydrated by oven (Marsilio et al., 2000) and they were marketed under refrigeration. Consequently, salt-drying technology gives rise to dehydrated olives with pH lower than 5.5 units unlike oven-drying olives with pH higher than 6 units unless the starting fruits are naturally fermented olives with pH lower than 4 units.

The water activity is another hurdle used by food processors to control the growth of pathogens. This parameter currently decreases from 0.98 to 0.99 in harvested olives to 0.92-0.85 in dry-salted olives as a consequence of both the reduction of moisture in the fruit and its increase in NaCl (Panagou et al., 2006; Ramírez et al., 2013). However, there are no data available on $a_w$ in commercial dehydrated olives. The mean value of $a_w$ in the analyzed samples was 0.85 (Table 3) although it ranged between 0.72 and 0.96, the latter value corresponding to sample 6 that contained Spanish olives, probably fermented before dehydration due to their low pH (3.95 units). It has been indicated that *C. botulinum* does not grow at $a_w$ lower than 0.94 (Wareing & Fernandes, 2007) so most of the commercial olives could comply with this limit although the growth of this microorganism has also been reported at $a_w$ as low as 0.85 in some cases (Kim & Foegeding, 1993). In addition, we found a great $a_w$ variability among individual olives from the same sample.

As expected, the NaCl content in these olives was high (Table 3) but it depended on how this content was calculated. First, we analyzed the NaCl content in the olives by mixing the olive pulp with distilled water, and the results were referred to as g of NaCl/100 g pulp, as has been reported in previous works (Değirmencioğlu et al., 2014; Panagou et al., 2006). In doing that, eight out of the 20 samples had a NaCl content lower than 10% established by the IOC (2004). However, both the IOC (2004) and the Codex Alimentarius, 2013 standards indicate that the NaCl content must be determined in the olive juice. As it is not easy to analyze the NaCl content in the olive juice, we referred the data from the mixture pulp/distilled water to the content in the olive juice taking into account the moisture of the olives (Table 3). Then, only samples 6 and 9 had a NaCl content in the olive juice lower than 10%, although the pH of sample 6 was 3.95 and $a_w$ of sample 9 was 0.93, so all the commercial dehydrated olives could be considered as microbiologically safe considering the pH/NaCl concentration/$a_w$ limits recommended for the inhibition of *C. botulinum* growth (Kim & Foegeding, 1993; Wareing & Fernandes, 2007). Also, the preservative sorbic acid was detected in sample 9 although it was not declared on label. Moreover, processors should think in the future about decreasing the concentration of NaCl in this product as demanded by consumers in combination with a reduction of the moisture in order to achieve low $a_w$. Although the average moisture in the analyzed olives was around 35%, there were samples (4, 9 and 15) with moisture close to 50%. A high moisture content in the olive means high $a_w$, so molds could grow during the shelf life of the product unless pasteurization was applied. If processors wants to reduce the NaCl content in the dehydrated olives in the future they should take care about the final moisture of the olives.

Besides moisture, another important component of dehydrated olives is the fat (Table 4) that ranged from 26% (sample 9) to 61% (sample 11) with an average value of 40%. Obviously, the fatty acid composition in the fat of theses olives can be comparable to that of olive oil rich in monounsaturated fatty acid, although it depends on the cultivar (Lanza et al., 2014). The average peroxide value of the commercial dehydrated olives (10.2 meq $O_2$/kg oil) was within the limit required for olive oils (EC Regulation,) and rather similar to the values reported for other table olive elaborations (Lanza et al., 2014; López-López et al., 2022; Pasqualone et al., 2014). On the contrary, the parameters associated with secondary oxidation ($K_{232}$ and $K_{270}$) were higher than the limits established for olive oils (EC Regulation,) and found in directly brined table olives (Lanza et al., 2014; López-López et al., 2022) but similar to those reported for green and black processed olives (Ruíz-Méndez et al.,

**Table 3**
Chemical characteristics of commercial dry black olives.

| Sample | pH | $a_w$ | Pulp moisture (%) | NaCl (%)[a] | NaCl (%)[b] |
|---|---|---|---|---|---|
| 1 | 4.58[c] | 0.88 | 33 | 8.6 | 17.5 |
| 2 | 3.90 | 0.86 | 27 | 9.4 | 22.1 |
| 3 | 4.90 | 0.82 | 38 | 12.8 | 23.2 |
| 4 | 5.35 | 0.91 | 48 | 8.4 | 12.9 |
| 5 | 4.42 | 0.79 | 23 | 10.2 | 27.6 |
| 6 | 3.95 | 0.96 | 43 | 4.8 | 7.8 |
| 7 | 4.67 | 0.85 | 22 | 6.8 | 19.1 |
| 8 | 5.08 | 0.81 | 40 | 13.0 | 22.8 |
| 9 | 5.40 | 0.93 | 51 | 6.6 | 9.8 |
| 10 | 3.65 | 0.73 | 16 | 20.8 | saturated |
| 11 | 3.30 | 0.72 | 12 | 17.2 | saturated |
| 12 | 4.47 | 0.90 | 38 | 10.0 | 18.0 |
| 13 | 4.65 | 0.82 | 38 | 13.0 | 23.5 |
| 14 | 6.10 | 0.91 | 38 | 8.0 | 12.4 |
| 15 | 5.25 | 0.87 | 49 | 11.6 | 17.7 |
| 16 | 5.60 | 0.87 | 43 | 10.0 | 16.5 |
| 17 | 4.95 | 0.82 | 34 | 10.2 | 19.9 |
| 18 | 5.44 | 0.85 | 42 | 11.0 | 18.5 |
| 19 | 4.35 | 0.89 | 31 | 7.0 | 14.8 |
| 20 | 4.66 | 0.75 | 24 | 13.6 | saturated |

[a] Analyzed in the mixture pulp:distilled water (1:1), and expressed as g NaCl/100 g pulp.
[b] Content referred to the olive juice, and expressed as g NaCl/100 mL juice.
[c] Standard deviations of pH, $a_w$, pulp moisture and NaCl of duplicate samples were always lower than 0.03, 0.02, 1.0 and 0.3 respectively.

**Table 4**
Oil quality indexes in commercial dry black olives.

| Sample | % Oil in pulp | $K_{232}$ | $K_{270}$ | $\Delta K$ | Peroxide value (meq $O_2$/kg oil) |
|---|---|---|---|---|---|
| 1 | 41[a] | 4.34 | 0.18 | 0.009 | 16.1 |
| 2 | 48 | 5.54 | 0.29 | 0.009 | 13.2 |
| 3 | 34 | 5.73 | 0.54 | 0.031 | 3.4 |
| 4 | 32 | 5.07 | 0.58 | 0.033 | 3.5 |
| 5 | 49 | 6.02 | 0.71 | 0.048 | 3.1 |
| 6 | 40 | 3.89 | 0.17 | 0.005 | 2.7 |
| 7 | 52 | 3.73 | 0.40 | 0.017 | 8.8 |
| 8 | 31 | 4.45 | 1.12 | 0.061 | 7.0 |
| 9 | 26 | 3.19 | 0.58 | 0.014 | 3.4 |
| 10 | 54 | 3.95 | 0.36 | 0.008 | 7.1 |
| 11 | 61 | 3.47 | 0.33 | 0.006 | 21.1 |
| 12 | 42 | 2.58 | 0.47 | 0.021 | 5.3 |
| 13 | 34 | 5.09 | 0.61 | 0.035 | 33.2 |
| 14 | 34 | 6.82 | 0.79 | 0.026 | 32.0 |
| 15 | 29 | 4.89 | 1.18 | 0.045 | 12.6 |
| 16 | 33 | 3.77 | 0.45 | 0.010 | 4.4 |
| 17 | 36 | 4.14 | 0.82 | 0.025 | 5.0 |
| 18 | 33 | 3.59 | 0.45 | 0.013 | 5.0 |
| 19 | 41 | 3.12 | 0.26 | 0.006 | 13.2 |
| 20 | 47 | 4.62 | 1.11 | 0.031 | 4.0 |

[a] Standards deviations of % oil, K232, K270, $\Delta K$ and peroxide value of duplicate samples were always lower than 1.0, 0.03, 0.02, 0.003 and 1.2 respectively.

2008). Bearing in mind that the analyzed dehydrated olives are marketed and three of the authors did not find rancidity sensation from these olives when open the containers, it seems that these high fat quality parameters cannot be associated with olive off-flavor.

The quality parameters of the olives (color and texture) were very similar for all the samples (Table 5). Dehydrated olives presented black color as reflected by their $L^*$, $a^*$, $b^*$ and $R_{700}$ values, except sample 6 that showed a higher $R_{700}$ value than the rest, corresponding with the dark brown color of these olives. As commented above, the olives from sample 6 were fermented before dehydration and they were enriched with the antioxidant ascorbic acid, as indicated on the label, so this substance will probably avoid an intense darkening of the fruit during the heating stage. Likewise, the firmness of these dehydrated olives presented an average of 26 N/fruit and standard deviation of 6.7 N/fruit, although this parameter ranged from 17.2 N/fruit to 42.6 N/fruit.

Among the microorganisms studied, yeast and molds were detected in 7 out of 20 samples (Table 2), although these microorganisms were found in a high population only in 4 of these samples (5, 7, 14 and 20), although it must be noted that no mold growth was observed on the surface of any sample. In fact, yeasts and molds have been the dominant microorganisms reported in packed dry-salted olives (Değirmencioğlu et al., 2014; Panagou, 2006). However, a high population of lactic acid bacteria has also been found in a few samples of commercial dry-salted olives (Panagou et al., 2006) and *Enterobacteriaceae* during the salting process of black olives (Ramírez et al., 2013). Among the 20 samples analyzed, the latter microorganisms were only detected in olives from sample 14. It is worth saying that a bulge container (jar and pouch) was not detected in any of the commercial samples despite the absence of sorbic acid in many of them (Table 1). Hence, the low percentage of oxygen and high of $CO_2$ in the inner atmosphere of most containers (Table 6) must be related to biochemical transformations that occur during olive storage more than to microbial growth, as has also been observed for black ripe olives packed in pouches after pasteurization (Romero et al., 2021). It must be noted that all samples were analyzed at least one month from packing on the arrival at the laboratory.

Gas atmosphere was analyzed on the arrival of the samples at the laboratory but they had been.

Besides fiber, monounsaturated fatty acids, vitamin E and minerals, table olives are a good source of bioactive compounds including phenolic compounds and triterpenic acids. The main phenolic compounds identified in the dehydrated black olives were hydroxytyrosol 4-*O*-glucoside, hydroxytyrosol and oleuropein (Table 7). It is well-known that the concentration of oleuropein decreases with olive ripeness while the content of hydroxytyrosol 4-*O*-glucoside increases (Romero et al., 2017). Also, the main transformation of these substances during the dehydration process is the enzymatic oxidation of *o*-diphenols, particularly oleuropein (Ramírez et al., 2013). Hence, hydroxytyrosol 4-*O*-glucoside was found in most of the analyzed samples except in samples 10 and 11, that were particularly rich in hydroxytyrosol due to the release of this substance from oleuropein during the fermentation stage prior to the dehydration process. It is worth mentioning that hydroxytyrosol is an ortho-diphenol, but it did not oxidize during heating due to the low pH achieved during olive fermentation in samples 10 and 11 (Table 3). Surprisingly, this substance was not found in sample 2 that corresponded to dry-salted olives obtained from non-fermented fruit. In particular, the level of hydroxytyrosol 4-*O*-glucoside was very high in samples 9 and 14, that were supposedly elaborated following the Italian Ferrandina method that includes the dehydration of the olives in an oven (Borzillo et al., 2000). Nevertheless, the concentration of this substance was also very high in samples 17, 13, 16 and 18 that were NaCl-cured, so this high content could be cultivar-dependent. Indeed, Throuba Thassos olives dehydrated by oven presented a very low content in oleuropein and most phenolic compounds (Mantzouridou & Tsimidou, 2011). By contrast, we found a high concentration of oleuropein in samples 5 and 20 corresponding to declared on label of Greek Throuba Tassos olives dehydrated by NaCl, which is in agreement with previous works (Melliou et al., 2015; Zoidou et al., 2010). However, sample 19 from Turkey also had a high level of this bitter compound and the cultivar was probably different to Throuba Tassos. Overall, the average concentration of phenolic compounds in the commercial samples analyzed was 2591 mg/kg, a much higher content than that reported for Spanish-style green and California-style black olives (Romero et al., 2004). This high level could be expected due to the elimination of the moisture during the dehydration process but the oxidation of these substances could also occur, particularly for *o*-diphenols at pH > 5.

Finally, it must be highlighted the high concentration of triterpenic acids found in dehydrated black olives, particularly maslinic acid (Table 7), the average content of total triterpenic acids and maslinic acid being 3578 mg/kg and 2487 mg/kg respectively. These substances are soluble in alkali so their concentration is currently lower in alkali treated olives than in directly brined olives (Alexandraki et al., 2014; Romero et al., 2010). Taking into account the range of concentrations reported for raw olives (1500–2000 mg/kg) (Romero et al., 2010), it seems that the dehydration process does not affect these substances so they can be found in high levels in commercial dehydrated black olives.

**Table 5**
Quality parameters of commercial dry black olives.

| Sample | Color parameters | | | | Firmness (N/fruit) |
|---|---|---|---|---|---|
| | $L^*$ | $a^*$ | $b^*$ | $R_{700}$ | |
| 1 | 18.3 (0.2)[a] | 1.3 (0.1) | 0.3 (0.1) | 5.0 (0.2) | 17.2 (1.3) |
| 2 | 18.9 (0.2) | 2.4 (0.2) | 1.2 (0.2) | 6.5 (0.2) | 18.7 (2.8) |
| 3 | 17.6 (0.1) | 0.7 (0.1) | −0.1 (0.0) | 3.4 (0.1) | 24.3 (0.7) |
| 4 | 18.3 (0.2) | 0.8 (0.0) | −0.1 (0.0) | 3.7 (0.1) | 30.2 (2.5) |
| 5 | 17.3 (0.2) | 1.5 (0.1) | 0.8 (0.1) | 4.6 (0.1) | 21.7 (0.3) |
| 6 | 20.6 (0.4) | 3.5 (0.2) | 2.3 (0.2) | 8.4 (0.5) | 17.7 (0.3) |
| 7 | 17.2 (0.4) | 1.1 (0.2) | 0.0 (0.0) | 4.5 (0.1) | 34.1 (0.5) |
| 8 | 17.6 (0.3) | 1.2 (0.1) | 0.4 (0.0) | 3.9 (0.1) | 26.5 (0.4) |
| 9 | 18.6 (0.1) | 0.8 (0.0) | −0.2 (0.0) | 4.0 (0.1) | 26.3 (2.7) |
| 10 | 17.3 (0.2) | 1.7 (0.1) | 0.3 (0.0) | 5.4 (0.1) | 25.4 (0.5) |
| 11 | 16.6 (0.4) | 1.5 (0.2) | 0.1 (0.0) | 5.4 (0.3) | 35.5 (0.5) |
| 12 | 19.4 (0.3) | 1.2 (0.2) | 0.5 (0.1) | 4.8 (0.2) | 27.0 (2.9) |
| 13 | 20.9 (0.5) | 0.9 (0.2) | 0.4 (0.1) | 4.2 (0.3) | 21.1 (0.4) |
| 14 | 19.4 (0.3) | 0.7 (0.1) | −0.2 (0.1) | 3.8 (0.1) | 29.5 (1.6) |
| 15 | 17.4 (0.2) | 1.3 (0.1) | 0.5 (0.1) | 3.8 (0.1) | 42.6 (2.6) |
| 16 | 18.0 (0.2) | 0.9 (0.1) | 0.1 (0.0) | 3.6 (0.1) | 19.1 (0.2) |
| 17 | 17.4 (0.1) | 0.7 (0.0) | −0.2 (0.0) | 3.7 (0.1) | 25.7 (0.7) |
| 18 | 17.4 (0.1) | 0.6 (0.0) | −0.2 (0.0) | 3.3 (0.1) | 21.2 (0.5) |
| 19 | 18.5 (0.2) | 1.4 (0.1) | 0.3 (0.1) | 5.6 (0.2) | 31.5 (1.5) |
| 20 | 16.1 (0.1) | 0.7 (0.0) | −0.3 (0.0) | 3.4 (0.1) | 33.0 (0.7) |

[a] Standard deviation of duplicate samples is shown between parenthesis.

**Table 6**
Atmosphere composition in the packing containers of commercial dry black olives.

| Sample | % $O_2$ | % $CO_2$ | % $N_2$ |
|---|---|---|---|
| 4 | 7.0 (0.3)[a] | 7.5 (0.4) | 85.5 (0.3) |
| 6 | 1.8 (0.4) | 9.1 (0.6) | 89.1 (0.5) |
| 8 | not detected | 39.1 (0.5) | 60.9 (0.5) |
| 9 | not detected | 47.1 (0.6) | 52.9 (0.6) |
| 10 | 5.1 (0.3) | 2.5 (0.3) | 92.4 (0.4) |
| 11 | 5.1 (0.5) | 2.5 (0.5) | 92.4 (0.4) |
| 12 | 2.6 (0.3) | 11.1 (0.2) | 86.3 (0.3) |
| 15 | 0.5 (0.2) | 18.0 (0.2) | 81.5 (0.5) |
| 16 | 2.3 (0.3) | 18.3 (0.3) | 79.4 (0.4) |
| 17 | not detected | 13.1 (0.6) | 86.9 (0.6) |
| 18 | not detected | 34.7 (1.2) | 65.3 (1.2) |
| 19 | not detected | 17.3 (0.9) | 82.7 (0.9) |
| 20 | 2.7 (0.2) | 8.5 (0.2) | 88.8 (0.2) |

[a] Standard deviation of duplicate samples is shown between parenthesis.

## 4. Conclusions

The results of this survey have disclosed great variability in

**Table 7**
Bioactive substances in the pulp of commercial dry black olives (mg/kg).

| Sample | Phenolic compounds | | | | Triterpenic acids | |
|---|---|---|---|---|---|---|
| | Hydroxytyrosol | Hy-4-Gl[a] | Oleuropein | Others[b] | Maslinic | Oleanolic |
| 1 | 126 (9) | 260 (16) | nd[c] | 211 (11) | 3469 (84)[d] | 1457 (71) |
| 2 | 270 (24) | nd[c] | nd | 332 (34) | 2178 (197) | 1287 (13) |
| 3 | 71 (6) | 1120 (38) | nd | 81 (20) | 1399 (1) | 716 (19) |
| 4 | 89 (52) | 1426 (82) | nd | 70 (15) | 852 (87) | 373 (22) |
| 5 | 87 (18) | 115 (37) | 712 (78) | 281 (12) | 2725 (107) | 1089 (862) |
| 6 | 686 (20) | 161 (22) | nd | 251 (5) | 3233 (287) | 1448 (1) |
| 7 | 99 (1) | 74 (11) | nd | 110 (8) | 3350 (62) | 1821 (36) |
| 8 | nd | 1760 (98) | nd | 259 (53) | 1885 (95) | 977 (29) |
| 9 | nd | 7677 (99) | nd | 194 (23) | 1326 (42) | 526 (37) |
| 10 | 3163 (36) | nd | nd | 325 (24) | 3841 (357) | 1204 (1) |
| 11 | 3021 (31) | nd | nd | 498 (9) | 3242 (309) | 1023 (152) |
| 12 | 198 (6) | 368 (22) | nd | 59 (30) | 5035 (128) | 2144 (34) |
| 13 | 173 (3) | 3934 (95) | nd | 90 (12) | 2598 (41) | 1190 (44) |
| 14 | nd | 6156 (78) | nd | 48 (23) | 1929 (235) | 873 (116) |
| 15 | 27 (3) | 599 (80) | nd | 26 (7) | 2014 (167) | 961 (95) |
| 16 | 119 (19) | 2304 (55) | nd | 60 (9) | 1185 (236) | 597 (24) |
| 17 | 153 (18) | 6297 (97) | nd | 65 (11) | 1131 (152) | 544 (38) |
| 18 | 61 (5) | 2924 (45) | nd | 70 (8) | 1846 (190) | 797 (103) |
| 19 | 406 (37) | 284 (12) | 989 (89) | 117 (8) | 3117 (163) | 1351 (151) |
| 20 | 213 (13) | 621 (78) | 1897 (87) | 31 (6) | 4385 (139) | 1440 (44) |

[a] Hydroxytyrosol 4-O-glucoside.
[b] Others is the sum of hydroxytyrosol glycol, tyrosol, vanillic and *p*-coumaric acids, luteolin-7-glucoside, luteolin and comselogoside.
[c] Not detected.
[d] Standard deviation of duplicate samples is shown between parenthesis.

physicochemical and microbiological characteristics among the different dehydrated black olives marketed worldwide. Almost all the samples were in line with the requirement (NaCl > 8–10% in the olive juice) established by IOC and CODEX Standards for dehydrated table olives. Indeed, most of the samples surpassed these limits and processors must think about reducing the NaCl content in these olives considering the current consumer demand for low sodium products. Moreover, if found, yeast and molds seem to be the predominant microorganisms in this product, so pasteurization must be the common practice for the elaboration of dehydrated black olives. Likewise, these olives are a very interesting nutritional food because they possess a high content of oil rich in monounsaturated fatty acids, around 40% of the olive pulp, more than 2500 mg/kg of phenolic compounds and are particularly rich in triterpenic acids that account for more than 3500 mg/kg. Hence, this is an in-demand product nowadays that must be adapted to the low sodium trend in foods because of the high NaCl detected in the samples analyzed. In addition, safety of the product must be assured by using pasteurization instead of preservatives, and dehydrated table olives must be valued in the future taking into consideration their high content in bioactive compounds.

## CRediT authorship contribution statement

**Pedro García-Serrano:** Conceptualization, Methodology, Formal analysis, Investigation. **Mercedes Brenes-Álvarez:** Investigation, Formal analysis. **Concepción Romero:** Investigation, Formal analysis. **Eduardo Medina:** Investigation, Formal analysis. **Pedro García-García:** Investigation, Formal analysis. **Manuel Brenes:** Conceptualization, Methodology, Writing – original draft, Writing – review & editing.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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