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**PACKING BLACK RIPE OLIVES IN RETORTABLE POUCHES WITH
DIFFERENT OXYGEN PERMEABILITY**

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ABSTRACT

The types of retortable pouches with different O₂ permeability and packing conditions (liquid, air or N₂ atmosphere), and processed with and without iron addition, have been studied. In all cases, consumption of oxygen and production of CO₂ were found during the first months of storage. Moreover, oxygen caused discoloration of the olives. Consequently, a loss of black color was observed during shelf-life of fruits packed under air atmosphere, particularly as the permeability of the pouch increased. Hence, the use of nitrogen atmosphere has been disclosed as a good method for packing ripe olives, especially in those pouches with high oxygen permeability. Likewise, the type of container and packing conditions did not influence the firmness of the olives but only the use of iron gave rise to firmer olives.

Key words: Nitrogen; Oxygen; Packing; Pouches; Ripe olives

30 ***Highlights***

- 31 - Oxygen gives rise to discoloration of packed black ripe olives
- 32 - Packing under nitrogen atmosphere can prevent olive discoloration
- 33 - Black olives must be packed in retortable pouches with low oxygen
- 34 permeability
- 35 - Olive firmness was not affected by the type of retortable pouch

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1. INTRODUCTION

Among the different types of table olives, the California-style black olives or black ripe olives are those whose production has steadily increased during the recent decades due mainly to their use for pizzas, salads and many other fast food products. These olives are very attractive to consumers because of their shiny black color. The elaboration processing involves a first stage of fruit preservation in an acidic solution (De Castro et al., 2007), followed by a darkening stage that consists of successive treatments (1-5) of the olives with dilute solutions of NaOH (lyes). During intervals between NaOH treatments, fruits are suspended in water through which air is bubbled (Sánchez-Gómez, García-García, & Rejano-Navarro, 2006). To prevent color deterioration, fruits are immersed in a ferrous solution (lactate or gluconate) for 6-24 hours (García, Brenes, Romero & Garrido, 2001; García, Romero & Brenes, 2018) and packed with a small amount of the ferrous salt. Finally, the product must be sterilized to ensure microbial safety (Sánchez-Gómez, García-García, & Rejano-Navarro, 2006).

Traditionally, these black ripe olives have been packed in jars or cans. However, consumers are demanding packaging that, once they reach their destination, occupies less space. To this end, plastic bags are an alternative to take into account. In addition, consumers prefer to see the black olives inside the packing container which, are hidden by the black color of the liquid, so table olive companies are demanding a product without the cover brine. Indeed, the cover brine is also a problem to get rid of once the containers are opened. Hence, plastic manufacturing companies have recently achieved retortable pouches that can be useful for packing black ripe olives.

There is only one work refers to the packing of black ripe olives in pouches submitted to a final sterilization step but it was used as a control of experiences of black ripe olives packed in pouches under acidic conditions (García, Brenes, Romero & Garrido, 1999). In this case, pasteurized and sterilized olives darkened due to the diffusion of oxygen inside the plastic pouches. This phenomenon has also been described in Spanish-style green olives packed in pasteurized pouches (Sánchez, López-López, Beato, De Castro & Montaña,

2017a), as the higher oxygen permeability of the pouch, the darker olives became (Sánchez et al., 2017b). Obviously, the permeability for oxygen of the films has a great influence on the color and most of the organoleptic characteristics, and it has been reported for peanuts and fruit juices (Vercelino Alves, Sarantopoulos, Segantini Saron & Bordin, 2001; Martín, Nepote & Grosso, 2016). Indeed, natural green olives packed in plastic pouches developed superficial browning rapidly during the pasteurization step (Piscopo, De Bruno, Zappia & Poiana, 2016).

Modified atmospheres have been widely explored to minimize effect of oxygen on food products packed in plastic pouches. Regarding table olives, the use of this technique has been studied for the packing in pouches of black dry-salted olives (Panagou, 2006; Değirmencioğlu, Gürbüz, Değirmencioğlu, Şahan & Özbey, 2011), natural black olives (Doulgeraki, Hondrodinou, Liopoulos, & Panagou, 2012) and black hot air-dried olives (Mantzouridou & Tsimidou, 2011). All these works were mainly focused on the microbial stability of the product and none have been performed on California-style black olives.

Nowadays, consumers are demanding food products without additives and importers of some countries are requesting black ripe olives without the addition of the ferrous salt. However, the black color of the olives fades when the iron salt is not added (García et al., 2001), so packing this product in pouches permeable to oxygen could reduce this problem.

According to the possible advantages that the use of plastic pouches could have for the packaging of black ripe olives, the aim of the present work was to determine the best type of film and packing conditions (liquid, air or controlled atmosphere) in order to maintain the characteristics of the traditional product.

2. MATERIALS AND METHODS

2.1. Black ripe olives

Fruits of the Hojiblanca cultivar (*Olea europaea* L.) stored for a period of two months in industrial tanks at the Agro Sevilla SCA factory under acidic

conditions were used for the assays (De Castro et al., 2007). Olives (5 kg) were put in two cylindrical oxidation chambers and immersed in 5 L of a NaOH solution (30 g/L) until it penetrated to the stone (Brenes, Romero, & García-García, 2017). Subsequently, the lye was removed and the olives were covered with 5 L of tap water for 20 hours. After draining, the fruits were put in a fresh tap water for 24 h while the pH of the liquid was maintained at 8.0 units by adding CO₂ to neutralize the excess NaOH in the olive flesh. Air was bubbled into the oxidation chambers throughout the whole process.

Oxidized olives from one of the oxidation chambers were packed immediately (product without iron addition), and the fruits of the other oxidation chamber were immersed in a ferrous gluconate solution (1 g/L) for 6 h to fix the black color formed (traditional process).

2.2. Packing conditions

Black ripe olives were packed in glass jars of 314 mL volume (JUVASA, Dos Hermanas, Spain) and three types of plastic complex pouches (SPgroup, Villarrubia, Spain): (i) Polyester and polypropylene (PET+PP G) with an O₂ permeability < 82 cm³/m²/day; (ii) Biaxially oriented polyamide and polypropylene (OPA STE+PP G) with an O₂ permeability < 28 cm³/m²/day) and (iii) High barrier polyester and polypropylene (PET PVDC PAST+PP G) with an O₂ permeability <10 cm³/m²/day. The technical characteristics of the films are shown in Table 1S.

An amount of 175 and 200 g of olives were put into the glass jars and plastic pouches, and covered with 145 and 200 mL of cover brine (40 g NaCl/L) respectively. When olives were processed traditionally with a ferrous gluconate fixation stage, the packing cover brine also contained 0.15 g/L of ferrous gluconate.

When cover brine was not used, the internal atmospheres of the jars and pouches were 150 and 145 mL respectively. **The inner atmosphere of the jars was always air and in the pouches was air or only nitrogen.**

Pouches were sealed first with a Tecnotrip sealer mod EVT-7-G-TD-SD (Terrasa, Spain) at 80 °C, and the bags were subsequently sealed again at 160° C in an Audion Elektro sealer equipment (Weesp, Holland) to support sterilization temperature.

2.3. Sterilization and storage

Both jars and pouches were sterilized at 121 °C in a computer-controlled Steriflow retort (Madinex, Barcelona, Spain) for the time needed to achieve an accumulated sterility of 15 F₀ in each container. Temperature inside the jars and pouches during sterilization was controlled with cylindrical programmable thermometers (Thermo buttons) of 17 mm diameter and 5 mm height (Thermotrack PC, Progres-Plus, France). It must be noted that these Thermo buttons put inside the containers were very small and similar in size the olives.

Finally, all containers were stored at 25 °C in a controlled temperature room for 5 months.

2.4. Experimental design

Olives darkened by the color fixation stage (Fe addition) were packed in glass jars and plastic pouches with (i) cover brine containing ferrous gluconate, and (ii) under air atmosphere without any packing liquid.

Olives darkened without the color fixation stage (without Fe) were also packed in jars and pouches with (i) NaCl solution, (ii) air atmosphere, and (iii) N₂ atmosphere. In addition, 4 glass jars were filled with only 30 g of these olives instead of the current 175 g under air atmosphere.

Two containers of each treatment were analyzed at 1 and 5 months after packing for chemical, superficial color and firmness parameters.

2.5. Analyses

The concentration of NaCl and the pH in the cover brine were measured according to traditional methods for table olives (Brenes, Romero & García, 2017).

The variation in the weight of the olives during their shelf life was carried out by weighing the drained fruits before packing and after 5 months.

The surface color of the fruits was measured by using a BYK-Gadner Model 9000 Color view spectrophotometer (Silver Spring, MD, USA) and it was expressed as reflectance at 700 nm (R_{700}). Lower reflectance values indicate darker fruit. Any interference from stray light was minimized by covering the samples with a box, which had a matte black interior. Results were the mean of 10 determinations.

Firmness was measured using a Kramer shear compression cell coupled to a TA.TXplus Texture Analyser (Stable Micro System, Surrey, UK). The cross-head speed was 200 mm/min. The machine measures the shear compression force in Newtons (N) to break 3 pitted olives. The value was the mean of 8 measurements.

Concentrations of O₂ and CO₂ in the inner atmosphere of packaging were measured with a Gaspac Advance Headspace/MAP Analyser (Illinois Instruments, Inc. Johnsburg, IL, USA) by introducing a hypodermic needle into plastic pouches or a steel piercing head into the metallic closure of glass jars.

Dissolved O₂ concentration in the cover brine was analyzed by introducing the probe HI98193 Dissolved Oxygen (Hanna Instruments, Eibar, Spain) in a beaker with about 30 mL of cover brine taking care that it does not aerate in the transfer from the container.

2.6. Statistical analyses

Statistica software 7.0 was used for data processing (Statistical for Windows, Tulsa, OK, USA). Comparison between means was made with Duncan's multiple range test and the differences were considered significant when $p < 0.05$.

2. RESULTS AND DISCUSSION

2.1. Sterilization conditions

Firstly, a sterilization program was developed for each container (pouch or glass jar) and packaging conditions (with liquid and without liquid) to reach an accumulated lethality of 15 units ($F_0=15$). As is shown in Figure 1A, the lack of cover brine delayed heat penetration inside the pouch, therefore that time of sterilization was prolonged in the retort so that all the olives had the same units of accumulated lethality (Figure 1B). In addition, the time needed to sterilize olives in jars was also higher than in pouches due to the greater thermal inertia of the glass (Figure 1S).

According to the data shown in Figures 1 and 1S, maintenance time at 121 °C was 13 min for pouches, and 15 min for glass jars both with cover brine. In packaging without liquid, 32 min and 44 min were needed to achieve the same accumulated lethality ($F_0=15$) for pouches and glass jars respectively.

2.2. Packing with cover brine

The initial concentration of oxygen in the cover brines (6-7 mg/L) diminished in all brines of packed olives after one month of storage regardless of the type of container (Table 1). However, lower concentrations were detected in the brines of the jars than in the pouches. After 5 months of storage, the oxygen almost disappeared in the brines of jars containing olives processed with and without iron and this effect also occurred in the pouches with the lowest oxygen permeability ($<10 \text{ cm}^3/\text{m}^2/\text{day}$). By contrast, the concentration of this gas increased or remained constant after 5 months of storage in the brines of olives packed in high oxygen permeability pouches ($<82 \text{ cm}^3/\text{m}^2/\text{day}$), processed with and without iron. These data indicate that oxygen was **depleted** during the shelf-life of olives packed in containers such as jars and pouches with low oxygen permeability, but it was not exhausted in pouches with high oxygen permeability because this gas must penetrate inside the bag from the environment.

The surface color of the traditional product with color fixation stage and addition of ferrous gluconate in the cover brine was very similar at 1 and 5 months for all containers with small statistically significant differences (Table 1). Black ripe olives (with ferrous salt addition) of Hojiblanca cultivar showed a slight increase in the reflectance at 700 nm (R_{700}) during their shelf-life for 3 years (García, Sánchez & Garrido, 2014a) therefore it could be expected not to find discoloration of the olives for the time studied.

It is well-known that olives color fades after sterilization (higher R_{700}), particularly if they are not processed with iron (Romero, García, Brenes & Garrido, 1995), and it explains the higher reflectance values of the olives processed without iron than with an iron fixation stage (Table 1). Besides, the differences among the types of packaging were small (less than 1.2 units of R_{700}).

Regarding firmness, the type of container (jar or pouch) did not have any effect on this parameter. Indeed, firmness of olives remained almost constant for the 5 months of storage. It must be noted that this product only loses around 20-30 % of its initial firmness after 3 years of shelf life (García et al., 2014a). However, firmness of olives processed and packed with iron was significantly higher than those without iron, with values at around 150-160 N/3 pitted olives and 138-142 N/3 pitted olives respectively.

Among the different steps for processing black ripe olives, the sterilization stage is determinant for the maintenance of the firmness of olives (Romero et al., 1995). The addition of divalent metals such as calcium in the packing cover brine or previously during the conservation stage (García Romero, & Brenes, 2014b) prevented loss of firmness of the olives. According to this, the addition of Fe^{++} avoided in a certain proportion the softening of the olives during the sterilization, as has been demonstrated for the Ca^{++} ion.

Finally, it has been reported that black ripe olives undergo a small loss of weight during packaging and sterilization, keeping the weight constant during the shelf life (García, Romero, & Brenes, 2014a). In these experiences, the behavior was the same, decreasing the weight of the fruits by an average of 1% regardless of the type of container and the time of analysis.

2.3. Packing without cover brine

2.3.1. Air atmosphere

During sterilization and the storage time, a disappearance of the initial oxygen concentration (21%) in the inner atmosphere of the glass jars was observed both in the packaging with olives processed with color fixation stage and when this process was not carried out (Figure 2). This oxygen consumption was also observed in the brines of olives packed with cover liquid in the same container (Table 1). In parallel, an initial increase in the CO₂ concentration in the inner atmosphere of the jars occurred (Figure 2S). There is not any explanation for this phenomenon because sterilization kills all microorganisms inside the jar and respiration of olives currently finishes after the first month of the olive storage stage before the darkening process (Romero, Brenes, García & Garrido 1996).

In the case of pouches, at the first month of storage the decrease of O₂ concentration in the internal atmosphere was lower than in the jars (Figure 2), which may be due to the permeability of the films. A graduation between the concentration of oxygen and the permeability of the films was also observed, the bag with less permeability having the lowest values of oxygen (<10 cm³/m²/day). Moreover, an increase in the concentration of O₂ took place in most bags after 5 months storage, tending to equalize with the outside atmosphere (21% O₂).

As happened in jars, an increase of the CO₂ concentration from the initial value of 0.03 % in the air occurred inside the pouches (Figure 2S) after one month of storage although this decreased with time of storage which was more pronounced as the permeability of the film was greater. This is due to the high CO₂ permeability of the films (Table 1S) and the difference in concentration between the inside of the pouches and the outside atmosphere.

Therefore, there were two very different behaviors according to the type of container: (i) in the jars, there was a gradual consumption of oxygen until its final consumption while (ii) in the pouches, there was an initial decrease that

was later compensated by the oxygen that penetrated inside, faster as the film was more permeable.

It has been reported that black ripe olives packed with liquid in cans and jars fades slightly during their shelf-life (García et al., 2014a). This phenomenon was observed in olives packed in jars without cover brine with air atmosphere regardless of processing with and without iron (Figure 3). Surprisingly, it also happened in olives packed in pouches with the 3 types of studied films, and it was more intense when iron addition was not performed in which case there was a graduation to higher values of R_{700} as the greater permeability of the film was observed.

It must be noted that an excess of oxygen (Figure 2) led to discoloration of black ripe olives (Figure 3), which was also observed by García, Brenes & Garrido (1991) during the oxidation process of the olives but not during packaging.

According to this, the presence of oxygen implies a loss of color during the shelf life in the packaging without cover brine.

To confirm this hypothesis, different amounts of olives were packed in jars without liquid, (i) full (175 g), having an air volume of 145 mL, and (ii) only 30 g of olives and 290 mL of air. At one month of storage, in the inner atmosphere of the jar full of olives, the O_2 concentration decreased to 3.95% (Figure 4A) producing a discoloration of the olives from a value of R_{700} before packing of 5.1% until reaching 6.9% (Figure 4B). In the jars with a low amount of fruit, as there was more volume of internal atmosphere, the concentration of oxygen decreased less (up to 17.1%) and discolored more (higher R_{700} value, 8.6%) because there was more available oxygen. Discoloration and disappearance of oxygen continued with the storage time.

Taking into account the initial concentration of oxygen in the packing atmosphere, the volume of gas and the amount of olives in the jars, the oxygen consumed per unit of fruit weight during the storage time was calculated, and it is depicted in Figure 4C. The higher consumption of oxygen the lighter the color of the olives achieved (higher percentage of reflectance).

2.3.2. N₂ atmosphere

Taking into account the former results, the product was packed in pouches under N₂ atmosphere. As can be seen in Figure 5A, black ripe olives without ferrous gluconate addition and packaged in pouches with an oxygen permeability lower than 28 cm³/m²/day retained their color for 5 months, whereas pouches with higher permeability (82 cm³/m²/day) gave rise to slight olive discoloration as well as a significant increase in oxygen content in the inner atmosphere of the pouch (Figure 5B). Indeed, the superficial color of these olives was darker (less R_{700}) than those without iron addition packaged in air (Figure 3) or with cover brine (Table 1) after 5 months of storage.

Therefore, the packaging of black ripe olives in pouches under N₂ atmosphere could be a good way to elaborate this product without the use of iron, which is demanded nowadays by many consumer countries.

2.3.3. Firmness and weight loss

With respect to firmness, neither the type of container (pouch or jar) nor gas atmosphere (air or nitrogen) influenced to a large extent the firmness of the final product even after 5 months of storage (Figure 6). As happened during the packaging with cover brine (Table 1), the only differential factor was the processing with iron addition that caused higher firmness of olives.

The loss of olive weight after sterilization and shelf-life must be highlighted. It is known that olives packed with cover brine lose around 1-2 % of the initial weight during sterilization and their shelf-life, which is a low percentage. In the case of olives packed without cover brine, a weight loss of around 2% was found after sterilization that even reached up to 5 % after 5 months of storage. This loss of weight led to the appearance of a small volume of liquid in the pouch.

3. CONCLUSIONS

After packing and sterilization, black ripe olives tend to consume oxygen and produce CO₂. Consequently, some changes occur during the shelf-life of this product. In the case of olives with and without iron addition packed in jars with

cover brine the color of the product undergoes slight changes after 5 months of storage, and a similar behavior occurred in retortable pouches with an oxygen permeability of films lower than $82 \text{ cm}^3/\text{m}^2/\text{day}$.

In contrast, it has been demonstrated that the olives packed without cover brine in air atmosphere suffer a discoloration during the shelf-life. To avoid this, they should be packed in pouches with low oxygen permeability in nitrogen atmosphere.

Packaging in plastic bags does not produce loss of texture over time and it was only noted that the presence of iron slightly increased the firmness of the final product. All these results must be taken into account in the future to make new black ripe olive products packed in retortable pouches.

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Legends of figures

Figure 1. Evolution of temperature in the retort and inside the plastic pouches during sterilization of ripe olives (A) to reach an accumulated sterility of 15 F_0 (B). Olives were packed with and without cover brine.

Figure 2. Concentration of oxygen (%) in the inner atmosphere of jars and pouches with an oxygen permeability lower than 10, 28 and 82 $\text{cm}^2/\text{m}^2/\text{day}$ containing black ripe olives processed with and without iron. Olives were packed without cover brine under air atmosphere and the analyses were carried out after 1 and 5 months of packing.

Figure 3. Superficial color (R_{700}) of black ripe olives processed with and without iron and packed in jars and pouches with an oxygen permeability lower than 10, 28 and 82 $\text{cm}^2/\text{m}^2/\text{day}$ under air atmosphere. The analyses were carried out after 1 and 5 months of packing.

Figure 4. Concentration of oxygen (%) in the inner atmosphere, superficial color (R_{700}) of olives and consumed O_2 (mMol O_2 /mg olive) during sterilization and storage. Black ripe olives were processed without iron and packed in jars with different amount of olives under air atmosphere. The analyses were carried out after 1 and 5 months of packing.

Figure 5. Influence of the oxygen permeability of pouch on the superficial color of black ripe olives (A) packed under nitrogen atmosphere and stored for 1 and 5 months at 25 °C. The concentration of oxygen (%) in the inner atmosphere of the pouch is also reported (B).

Figure 6. Firmness (N/3 pitted olives) of black ripe olives processed without iron and packed in pouches under N_2 and air atmosphere, and olives processed with iron and packed under air atmosphere. The analyses were carried out after 1 and 5 months of packing.

Figure 1S. Evolution of temperature in the retort and inside the glass jars during sterilization of ripe olives (A) to reach an accumulated sterility of 15 F_0 (B). Olives were packed with and without cover brine.

464

465 **Figure 2S.** Concentration of CO₂ (%) in the inner atmosphere of jars and
466 pouches with an oxygen permeability lower than 10, 28 and 82 cm²/m²/day
467 containing black ripe olives processed with and without iron. Olives were
468 packed without cover brine under air atmosphere and the analyses were carried
469 out after 1 and 5 months of packing.

470

Table 1. Influence of the type of packing container on the superficial color and firmness of black ripe olives processed with and without iron addition packed in cover brine. Oxygen concentration was also measured in the cover brine.

O ₂ permeability (cm ³ /m ² /day)	GLASS JARS		POUCHES			
			< 10		<82	
Time of storage (months)	1	5	1	5	1	5
<u>Processed and packing with Fe:</u>						
O ₂ concentration (mg/L)	0.25 (0.02) ^a b ^b	0.02 (0.02) a	1.13 (0.02) d	0.02 (0.1) a	0.66 (0.14) c	1.5 (0.14) e
Superficial Color (<i>R</i> ₇₀₀)	6.84 (0.29) b	6.33 (0.30) ab	6.77 (0.20) b	6.47 (0.22) ab	5.74 (0.38) a	6.39 (0.26) ab
Firmness (<i>N</i> /3 <i>pitted olives</i>)	151.7 (0.1) a	153.6 (1.4) a	156.2 (1.4) a	154.7 (3.4) a	161.0 (3.2) a	158.8 (3.5) a
<u>Processed and packing without Fe:</u>						
O ₂ concentration (mg/L)	0.28 (0.12) b	0.01 (0.0) a	1.13 (0.03) c	0.01 (0.07) a	1.16 (0.01) c	1.25 (0.0) c
Superficial Color (<i>R</i> ₇₀₀)	10.19 (0.05)ab	9.70 (0.28)a	11.36 (0.77)c	11.02 (0.28)bc	10.31 (0.19)ab	9.81 (0.44)a
Firmness (<i>N</i> /3 <i>pitted olives</i>)	144.7 (4.2) a	140.0 (3.8) a	140.2 (5.4) a	141.2 (4.3) a	138.7 (1.8) a	139.2 (5.1) a

Note: ^a Standard deviation of duplicate in parenthesis; ^b Row values followed by the same letter do not differ at the 5% level of significance according to Duncan's multiple-range test

Table 1S. Technical characteristics of the films to make the pouches used in the experiences of ripe olive packaging

TECHNICAL DATA	UNIT	DESCRIPTION OF COMPLEX			METHOD
		High barrier polyester and polypropylene	Biaxially oriented polyamide and polypropylene	Polyester and polypropylene	
		PET PVDC PAST+PP G	OPA STE+PP G	PET+PP G	
Oxygen permeability	cm ³ /m ² /day	<10	<28	<82	ASTM D 3985
Thickness	µm	107 ± 10%	109 ± 10%	106 ± 10%	ASTM E 252
Density	g/cm ³	0.99 ± 10%	0.98 ± 10%	0.95 ± 10%	ASTM D 1505
Unit weight	g/m ²	106.4 ± 10%	103.9 ± 10%	103.6 ± 10%	ASTM D 252
Water vapor permeability	g/m ² /day	<2.5	<3	<2.5	ASTM F 1249
Permeability to CO ₂	cm ³ /m ² /day	<39.5	<112.5	<326.5	ASTM D 3985
Permeability to N ₂	cm ³ /m ² /day	<2.5	<6	<16.5	ASTM D 3985
Departure sealing temperature	° C	150-160	150-160	150-160	SP GROUP 1 seg, 2 Bar

Figure 1

Figure 1S

Figure 2

Figure 2S

Figure 3

Figure 4

Figure 5

Figure 6