

Effect of vacuum impregnation on physical changes during table olive processing

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SUMMARY: Among the benefits which vacuum impregnation (VI) may provide to fruits and vegetables, this study focused on weight and texture changes during the processing of table olives. VI applied to Manzanilla olives led to around 10% weight gain, which was maintained after their packing as black olives. However, this weight gain was only around 4 % for Hojiblanca olives. Likewise, the use of calcium chloride was recommended to maintain the firmness of the olives, in particular those of the softer Manzanilla cultivar. With regard to the Spanish-style, the Hojiblanca cultivar achieved around 4% weight gain during processing but the use of VI for Manzanilla olives was ruled out due to softening of the fruit. In addition, the black and green color of olives and their flavor were not modified by the application of VI. This technology could be very useful to reduce weight loss during table olive processing.

KEYWORDS: *Calcium; Firmness; Table olive; Vacuum; Weight.*

RESUMEN: *Efecto de la impregnación al vacío sobre los cambios físicos durante la elaboración de aceitunas de mesa.* Entre los beneficios que la aplicación de la impregnación al vacío puede originar en frutas y verduras, este estudio se ha centrado sobre los cambios en la textura y el peso de las aceitunas de mesa durante su procesamiento. La impregnación al vacío de aceitunas Manzanilla dio lugar a un aumento cercano al 10 % de peso y ello se mantuvo después de su envasado como aceitunas negras, mientras que esta ganancia fue de sólo el 4% para aceitunas Hojiblanca. Asimismo, se vio necesario el empleo de cloruro cálcico para el mantenimiento de la textura de los frutos, en particular para aquellos de la variedad Manzanilla. Con respecto al procesamiento como verdes estilo español, la variedad Hojiblanca consiguió un aumento del 4% en peso debido al empleo de impregnación al vacío y este tratamiento se descartó para la Manzanilla debido al ablandamiento de la aceituna. Además, se debe indicar que el color verde y negro de las aceitunas no se vio afectado por el hecho de emplear impregnación al vacío. Esta tecnología podría ser muy útil para reducir las pérdidas de peso que se producen durante la elaboración de aceitunas de mesa.

PALABRAS CLAVE: *Aceitunas de mesa; Calcio; Firmeza; Peso; Vacío.*

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1. INTRODUCTION

Vacuum impregnation (VI) is a well-known technique which has been investigated in a wide range of fruits and vegetables (Fito *et al.*, 1996; Zhao and Xie, 2004; Blanda *et al.*, 2008; López and Moreira, 2019). Among the different applications of VI, it has been studied extensively in recent years for the elaboration of minimally processed and dehydrated fruits enriched with health-promoting substances including phenolic compounds (Luo *et al.*, 2019; Tylewicz *et al.*, 2019). In addition, the infusion of calcium and the enzyme pectin methylesterase into fruits via VI is carried out to increase their firmness (Banjongsinsiri *et al.*, 2004; Quintanilla *et al.*, 2018), although the use of VI without firming agents may induce fruit softening due to structural deformations (Tylewicz *et al.*, 2019). The rapid acidification of vegetables is another interesting application of VI to control microbial growth (Derossi *et al.*, 2011). With regard to table olives, VI has been studied for the enrichment of green olives with iron (Zunin *et al.*, 2017) and to accelerate the debittering stage with NaOH (Tamer *et al.*, 2013).

A consequence of the use of VI on fruits and vegetables is often an increase in their weight (Derossi *et al.*, 2010; Sirijariyawat *et al.*, 2012; Parreidt *et al.*, 2018). The application of a high vacuum level to plant tissue leads to the expansion of the gas trapped inside the intercellular spaces and capillaries, so an increase in the porosity volume of the tissue occurs. Hence, the penetration of the liquid inside the pores during the relaxation step of VI gives rise to an increase in the weight of the fruit (Schulze *et al.*, 2012; Tylewicz *et al.*, 2019). Among the many variables which can affect the VI phenomenon, the porosity fraction of the vegetable and fruit tissue is one of the most important. This parameter may range from 1.6 to 35.9% (Derossi *et al.*, 2012), and it has been estimated at around 4% in raw olives of the Hojiblanca cultivar (Romero *et al.*, 1996).

Changes in the weight of table olives currently occur during processing and this is of economic importance for industries. Although no reports are available on the changes in the weight of Spanish-style green olives, small weight increases occasionally occur during the debittering and fermentation stages of this product (private industrial communication). By contrast, weight loss has been reported during

the storage of olives of the Hojiblanca cultivar intended for processing as black ripe olives; whereas increase in weight takes place during the darkening stage of the elaboration of this commercial preparation (García *et al.*, 2014).

The aim of this work was to investigate the application of VI on raw olives which are then processed as Spanish-style green olives and black ripe olives, in particular its effect on weight changes during elaboration and its influence on the quality of the final product. The influence of calcium addition to prevent firmness loss was also assessed.

2. MATERIALS AND METHODS

2.1. Olive material

Olives of the Manzanilla and Hojiblanca cultivars were supplied at the end of September during the 2016/2017 olive season from local farmers located in the province of Seville (Spain). Three and four independent lots of olives of the Manzanilla and Hojiblanca cultivars were purchased, respectively. The fruits were harvested with green-yellow color on the surface and, on arrival at the laboratory, leaves and small branches were removed, and the olives were sorted in order to use those with uniform size (280–320 fruit/kg).

2.2. Assays of VI

The VI equipment consisted of a modified pressure cooker (10 L volume) with a pressure gauge connected to with a vacuum pump with a rubber tube (Millipore Corp.), which allowed adjustment of the level of vacuum. Before vacuum assays, the olives were submerged in tap water and the liquid was drained for five minutes. Subsequently, lots of 4 kg of wet olives were put inside the pressure cooker and immersed in 5 L tap water at room temperature. Except for the experiments aimed at studying the effect of the vacuum level, 0.8 bar vacuum was maintained during all assays for 15 minutes inside the chamber before the atmospheric pressure was restored. All samples were kept in tap water for a relaxation time of 15 minutes at atmospheric pressure. Afterwards, VI olives were processed as table olives.

Olives of the Manzanilla cultivar with different sizes ranging from 15 to 19 mm equatorial diameter were also vacuum impregnated to test the effect of olive size on weight changes.

2.3. Spanish-style green olive processing

For each of the four Manzanilla lots, 5 kg of vacuum-impregnated olives were put into 8.5 L cylindrical polyethylene vessels and covered with 3.4 L of 0.47 M NaOH solution until it penetrated two-thirds of the way to the pit of the olives (Figure 1). Subsequently, the olives were washed with tap water for seven hours and then covered with 10% (w/v) NaCl solution or the same brine spiked with 2 g/L CaCl_2 . Olives that were not submitted to VI were similarly debittered, washed and put into brine with and without calcium to use as controls. Finally, the fruits were left to spontaneous fermentation for eight months and the weight change was recorded (Figure 1). All assays were run in duplicate.

The four lots of Hojiblanca olives were processed as described above, except for the NaOH concentration, which was 0.52 M.

2.4. Black ripe olive processing

Vacuum-impregnated olives (5 kg) of the three lots of Manzanilla cultivar were put into the 8.5-L vessels and covered with 3.4 L of 1.5% acetic acid with and without 3 g/L CaCl_2 (De Castro *et al.*, 2007). The olives which were not submitted to vacuum impregnation were also stored in

similar acetic acid solutions (controls). All assays were run in duplicate. After eight months of storage, the fruits were darkened following the recently developed single lye treatment (Brenes *et al.*, 2017). Three kilograms of olives were put into four methacrylate horizontal cylindrical containers and covered with 3 L of a 0.63 M NaOH solution (lye) until the lye reached the pit (4h). Then the alkaline solution was removed and the olives were submerged in tap water for 24 h with aeration. This first washing water was drained and the fruits were covered with fresh tap water for another 24 h with aeration as well as the pH being controlled in the liquid below 8.0 units with the addition of CO_2 . Subsequently, they were covered with a 0.1% (w/v) ferrous gluconate solution for five hours to fix the black color which had formed. Finally, the stones were removed from the olives with an industrial pitting machine (OFM, Seville, Spain), and 145 g of pitted olives were bottled in A314 jars (Juvasa, Dos Hermanas, Spain) and covered with 175 mL of a solution containing 32 g/L NaCl and 0.2 g/L ferrous gluconate. The jars were sterilized and maintained at ambient temperature for two months before analyses (Figure 1).

The four lots of Hojiblanca olives were processed as described above, except for the NaOH concentration, which was 0.70 M.

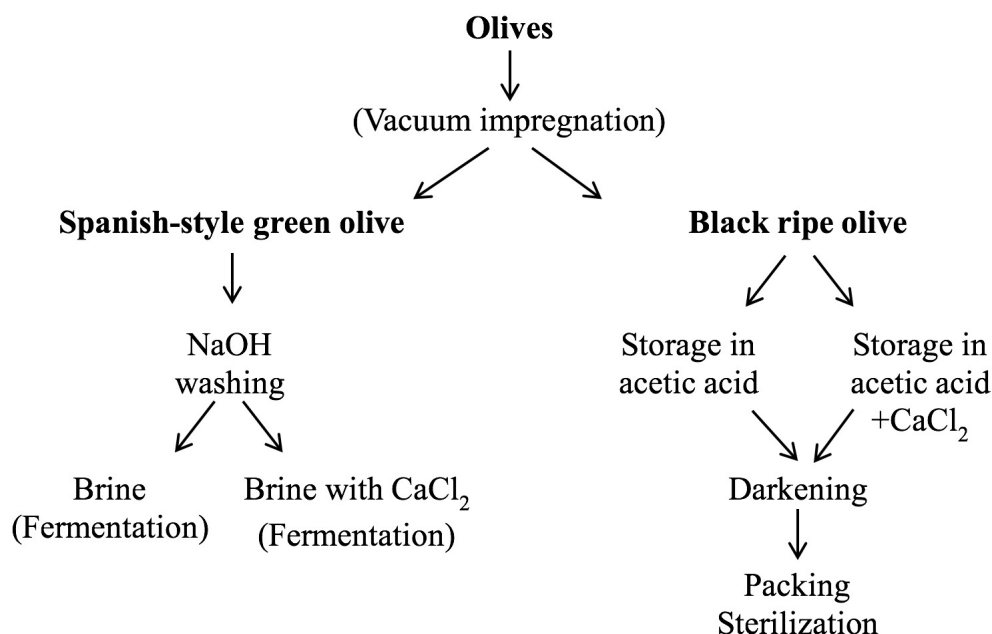


FIGURE 1. VI experimental design for olives of the Manzanilla and Hojiblanca cultivars.

2.5. Weight variation analysis

The percentage of weight change (DW) was calculated for every step of the olive processing as:

$$\Delta W = (W_i - W_f / W_i) \times 100$$

where W_i is the initial weight of the wet olives before the impregnation of processing (controls) and W_f is the final olive weight after each processing step.

Weight change was measured in duplicate for each of the three and four lots of Manzanilla and Hojiblanca cultivars, respectively.

2.6. Firmness analysis

The firmness of the fruits was determined using a Kramer shear compression cell coupled to a Texture Analyzer TA.TX plus (Stable Microsystems, Godalming, UK). The crosshead speed was 200 mm/min. Firmness was the mean of 10 replicate measurements, each of which was performed on three pitted olives, and expressed as Newton/100 g pitted olives.

2.7. Chemical analysis

The concentration of NaCl was analyzed by titration with a 0.1 N silver nitrate solution. The free acidity and pH of the brine were measured with a Metrohm 670 Titro processor (Herisau, Switzerland)

(De Castro *et al.*, 2007). Titratable acidity was determined by titrating to pH 8.3 with 0.2 M NaOH and expressed as lactic acid.

2.8. Statistical analyses

Statistical comparisons of the mean values for each experiment were carried out by one-way analysis of variance (ANOVA), followed by the Duncan's multiple range test ($p < 0.05$) using SPSS software v. 23.0 (IBM Corp., Armonk, NY).

3. RESULTS AND DISCUSSION

3.1. Effect of vacuum degree and olive size on weight changes

Figure 2 shows the effect of the vacuum level on the weight changes in Manzanilla olives during the application of VI at room temperature. A rather linear relationship between weight gain and the vacuum applied was found from 0.2 to 0.7 bars. Thereafter the rate of weight gain decreased up to 0.8 bars. Hence, 0.8 bars was the vacuum applied thereafter for all assays. In addition, preliminary experiments demonstrated that 15 min of vacuum application and relaxation were sufficient to reach the maximum effect. In the end, more than 10% weight gain was achieved in these olives of the Manzanilla cultivar impregnated with tap water. These results are in accordance with many others reported for fruit

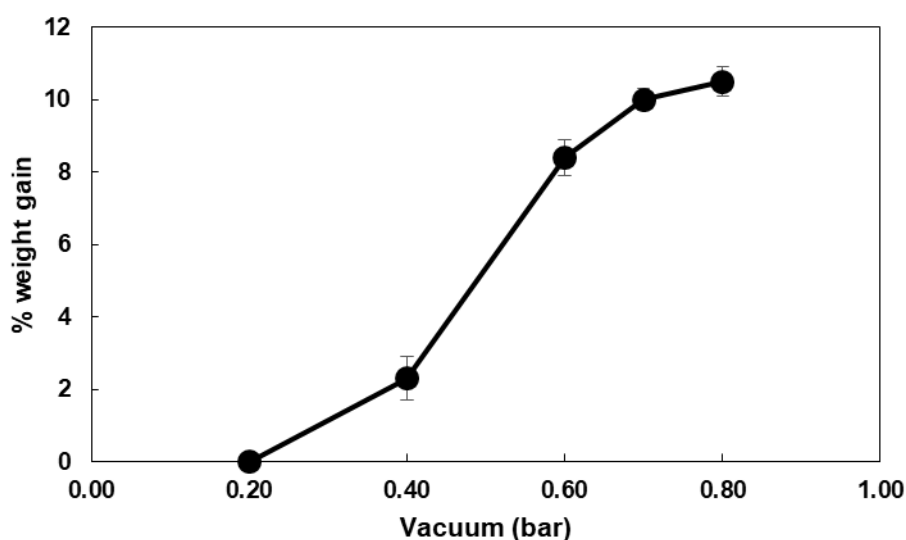


FIGURE 2. Influence of the degree of vacuum (bar) on the weight gain of olives of the Manzanilla cultivar (17 mm equatorial diameter).

and vegetables including apple slices (8-7% gain) (Mújica-Paz *et al.*, 2003; Parlawska *et al.*, 2019), peppers (4-11% gain) (Derossi *et al.*, 2010), mango (10-15% gain) (Sirijariyawat *et al.*, 2012), melon (4-8 % gain) and potatoes (8-11% gain) (López and Moreira, 2019; Parreidt *et al.*, 2019; Luo *et al.*, 2019). It is known that a high vacuum level increases the porosity of fruit tissue as a result of high expansion and release of the gas inside the pores of fruit but there is only one record regarding the intercellular volume of raw fruit of the Hojiblanca cultivar (4%) (Romero *et al.*, 1996). Hence, it could be assumed that VI led to deformation and expansion of the Manzanilla olive capillary with an increase in its volume and fruit weight (Figure 2). Because the effectiveness of VI could be influenced by olive size, the influence of this parameter was studied on olives of the Manzanilla cultivar, and the results obtained are depicted in Table 1: the greater the size of the

olive, the higher the weight gain. These findings also demonstrated that the volume of the fruit increased as a consequence of the VI treatment, so the caliber of the olives decreased to a large extent. Consequently, VI assays were run thereafter with olives of the same size.

3.2. Black ripe olives

First, it must be highlighted that olives of the Manzanilla and Hojiblanca cultivars which were vacuum-impregnated fermented during the storage period before the darkening stage in a similar manner to those that were not treated. In addition, the lye treatment lasted the same amount of time for both treated and non-treated olives.

The weight changes during the elaboration of the Manzanilla cultivar is depicted in Figure 3. All changes referred to the initial weight as indicated

TABLE 1. Influence of olive size on the physical characteristics of Manzanilla fruit submitted to VI (0.8 bar) for 15 minutes.

Equatorial diameter (mm)	Weight gain (%)	Volume gain (mL·kg ⁻¹)	Initial caliber (fruit·kg ⁻¹)	Caliber after VI (fruit·kg ⁻¹)
15	9.0	70	402	375
17	11.1	85	307	274
19	11.6	120	220	195

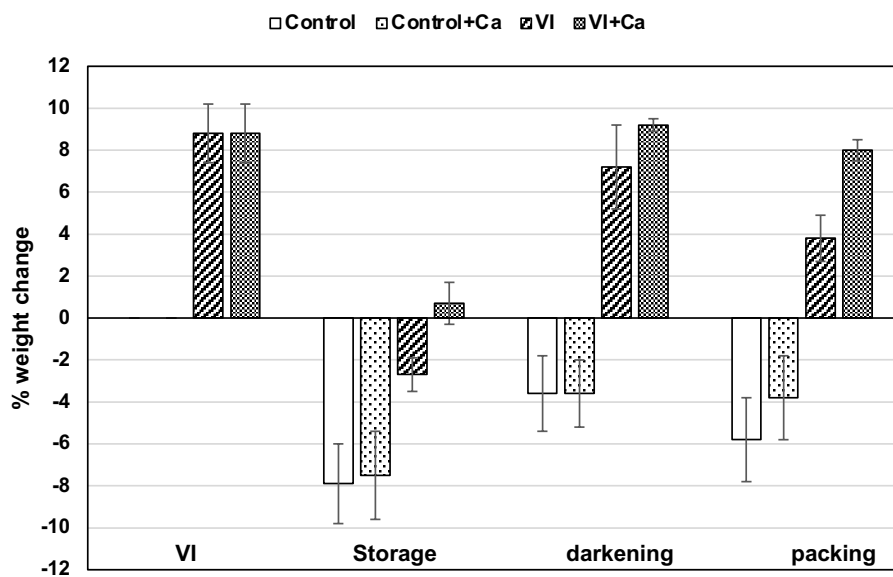


FIGURE 3. Effect of VI on the changes in olive weight of the Manzanilla cultivar during its processing as black ripe olives. Calcium chloride was employed during the storage stage. Bars are the mean value of three independent olive lots elaborated in duplicate, and they represent the accumulated gain or lost olive weight throughout the whole process. Standard deviation is depicted on the bars.

in section 2.5. The non-VI Manzanilla olives lost around 8% of their weight during their preservation stage in the acidified medium for eight months, which is a higher weight loss than previously reported for the Hojiblanca cultivar (García *et al.*, 2014). By contrast, VI olives reduced their weight by only around 2.8%, although the weight gain achieved during the VI application must be taken into account, thereby a difference of about 5.5% weight between control and VI olives after this preservation period occurred. Subsequently, stored fruits were submitted to the darkening stage and the olives gained weight, as expected from previous studies (García-Serrano *et al.*, 2020). However, weight gain was not the same for non-VI and VI olives. The former olives reduced their 8% loss obtained during the preservation stage to only 4% after the darkening stage; while the VI olives resulted in weight gain of around 7% after darkening so that the difference in weight between untreated and VI olives increased by up to 11%. Subsequently, the olives were packed, sterilized and analyzed after two months at room temperature and the difference was reduced to 9.8%.

It is well-known that the application of VI to fruit and vegetables may promote loss in firmness, in particular when using hypertonic solutions (Zhao and Xie, 2004). On the contrary, this technique has been widely tested to improve the texture of foods

by introducing either calcium or the enzyme pectin methylesterase into them (Quintanilla *et al.*, 2018; Servillo *et al.*, 2018). We tried to perform VI on olives with calcium chloride solution but the cation did not penetrate inside the fruit nor was any weight change noted (data not shown), so all our VI assays were run with only tap water. It must be noted that olive skin is a strong barrier against the diffusion of substances into the pulp of the fruit which counteracts the effectiveness of VI, which was observed for the application of VI during the alkaline treatment of the olives (Tamer *et al.*, 2013). Hence, taking into consideration previous studies on the improvement in texture with the addition of calcium to the liquid at the preservation stage (Brenes *et al.*, 1994; García-Serrano *et al.*, 2020), the storage solutions of both cultivars (Manzanilla and Hojiblanca) were also spiked with 3 g/L of calcium chloride (Figure 1). The influence of calcium on the weight changes in Manzanilla olives which were not treated with VI was not relevant (Figure 3). However, it was observed that the cation gave rise to higher weight gain in VI olives due to an almost 4% higher weight gain in VI packed olives with calcium added to the preservation liquid than in VI olives without calcium added (Figure 3). Additionally, calcium led to better texture in packed Manzanilla and Hojiblanca olives, regardless of the VI treatment (Figure 4).

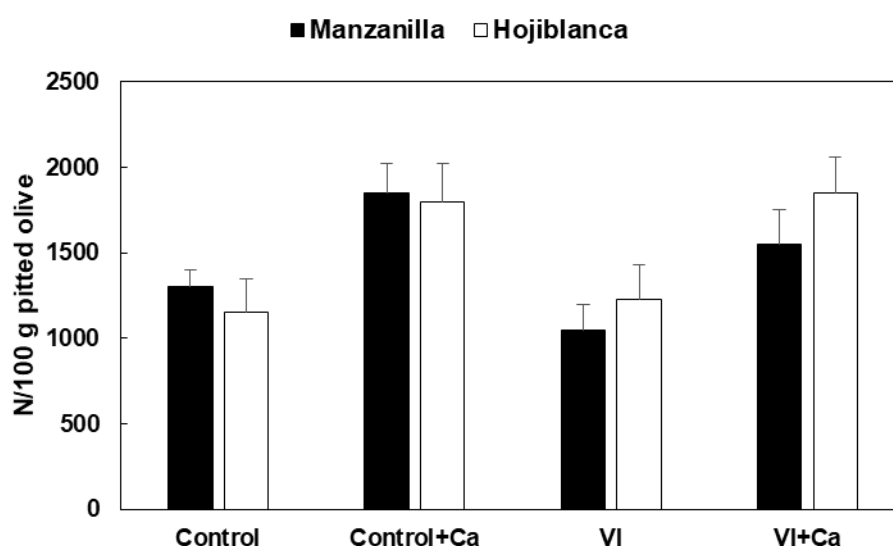


FIGURE 4. Influence of VI on the firmness of processed black ripe olives of the Manzanilla and Hojiblanca cultivars. Calcium chloride was employed during the storage stage. Standard deviation is depicted on the bars. Bars followed by a different letter indicate significant difference according to Duncan's test ($p < 0.05$).

In fact, slightly lower firmness in VI olives than untreated olives of the Manzanilla cultivar was achieved although it was not statistically significant; while the texture of packed Hojiblanca olives was not affected by the VI treatment (Figure 4).

The cell wall composition of the Manzanilla and Hojiblanca cultivars is very different (Jiménez, *et al.*, 1994), and the texture of the later cultivar is currently higher than the former (García-Serrano *et al.*, 2020), which could explain the lower weight changes found in the VI Hojiblanca cultivar during processing as black ripe olives (Figure 5) than in the VI Manzanilla (Figure 3). The application of VI to raw Hojiblanca olives only led to 4% weight gain in comparison to around 9% in Manzanilla fruit. In addition, weight changes were similar in all Hojiblanca olives after the storage period, regardless of VI treatment or calcium addition to the preservation solution (Figure 5). However, a weight gain of around 1% was detected after darkening in VI olives in comparison to around 2% loss in untreated olives, and these differences in weight even increased after packing, in particular when calcium was added to the preservation liquid so that around 4% higher weight was achieved in the VI Hojiblanca olives with calcium added than in untreated and non-added calcium olives. Any conclusion arising from these results can be explained by the impor-

tance of the olive cultivar in weight gain in the fruit after VI application and table olive elaboration.

It must also be noted that the color and flavor of the black olives were not affected by the use of VI on either the Manzanilla or Hojiblanca cultivar (data not shown). These quality parameters were evaluated by a sensory analysis carried out by 5 trained panelists.

3.3. Spanish-style green olives

The VI technique was also investigated for processing this commercial preparation (Figure 6). Unfortunately, the application of VI to Manzanilla olives softened the fruit to such a large extent after the alkaline treatment that it made them unmarketable (data not shown). In contrast, this softening effect was not observed for the Hojiblanca cultivar, which currently has a stronger texture than the Manzanilla, as mentioned above. In this case, VI Hojiblanca olives after fermentation presented around 6-7% weight gain while non-VI olives showed only around 2-3% weight gain. Again, the alkaline treatment led to an increase in the weight in both the control and VI olives which was reduced during the eight-month fermentation period. Thus, VI olives of the Hojiblanca cultivar had around 4% higher weight than

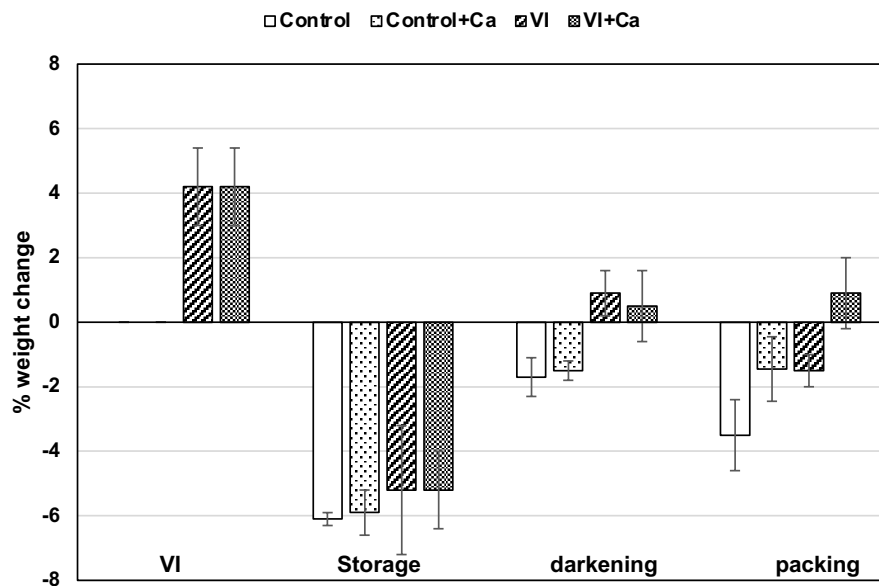


FIGURE 5. Effect of VI on the changes in olive weight of the Hojiblanca cultivar during its processing as black ripe olives. Calcium chloride was employed during the storage stage. Bars are the mean value of four independent olive lots elaborated in duplicate, and they represent the accumulated gain or lost olive weight throughout the whole process. Standard deviation is depicted on the bars.

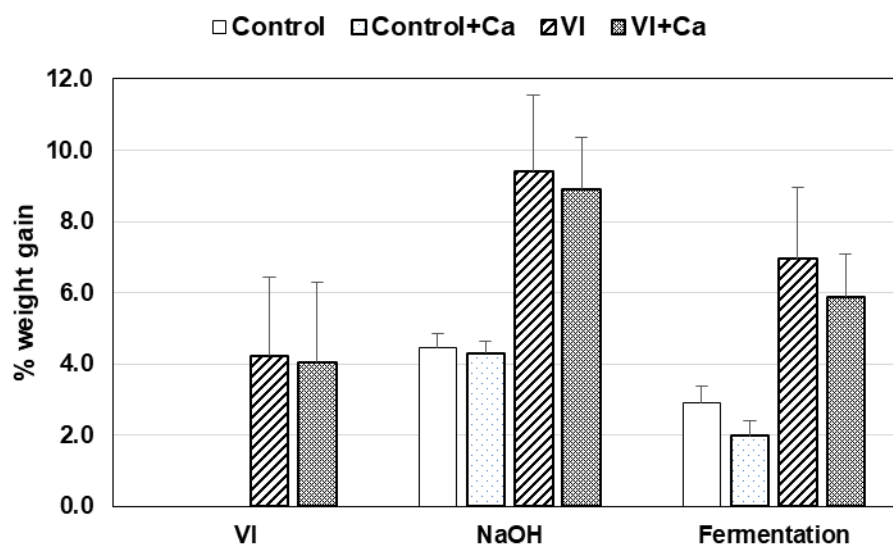


FIGURE 6. Influence of VI on the changes in olive weight of the Hojiblanca cultivar during its processing as Spanish-style green olives. Calcium chloride was added into the fermentation brine. Bars are the mean value of three independent olive lots elaborated in duplicate, and they represent the accumulated gain or lost olive weight throughout the whole process. Standard deviation is depicted on the bars. Bars followed by a different letter indicate significant difference according to Duncan's test ($p < 0.05$).

untreated olives after fermentation, regardless of the calcium addition.

Finally, it is worth noting that the lactic acid fermentation was similar in the VI olives and control and the final flavor and color was not affected by the use of this technique to the raw olives (data not shown). These quality parameters were also evaluated by sensory analysis carried out by 5 trained panelists.

4. CONCLUSIONS

It has been determined that the application of VI to raw olives can contribute to the reduction in weight loss or even increase in weight during the processing of table olives. This increase in weight was cultivar dependent, higher for the Manzanilla cultivar than Hojiblanca, which could probably be attributed to the different texture and cell wall composition of these two cultivars. It must be pointed out that olives gained weight during the VI treatment, but the application of a vacuum also favored a higher weight increase in olives during alkaline treatment than in untreated olives. Consequently, VI olives of the Manzanilla and Hojiblanca cultivars gained around 10 and 4% weight, respectively, in comparison to untreated olives processed as black ripe olives. In the case of Spanish-style green olives, VI was ruled out for the Manzanilla cultivar because the

firmness of the olives was too soft after the alkaline treatment. In contrast, the Hojiblanca cultivar could be processed following this elaboration method, and around 4% weight gain was found after the fermentation stage in comparison to untreated olives. In addition, the color and flavor of the olives were not affected by the use of VI. Therefore, VI seems a promising technique for processing table olives in order to reduce weight loss or even gain weight without loss in firmness in the final product, particularly with calcium addition.

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request

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