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Research Paper / Araştırma Makalesi

Effect of Prepackaging Treatments and Different Gas Composition on Physicochemical and Sensory Properties of *Hurma* Olive (*Erkence cv.*)

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ABSTRACT

Hurma olive is a special olive cultivar that loses its bitter taste before harvesting, and sweetness occurs when the fruit is still on the tree. Thus, it can be directly consumed right after being harvested. Studies on the preservation of *Hurma* olive without salt have been very limited and it has not been implemented in practice yet. In this study, some quality characteristics of *Hurma* olives under different modified atmospheres were determined during storage. For this purpose, the efficacy of washing, lactic acid dip, and modified atmosphere packaging (MAP) were studied throughout 120 days at 1°C. *Hurma* olive samples were washed with tap water and dipped in lactic acid solution (0.2% w/v) for 1 minute before packaging. After the application of vacuum, samples were packaged under the atmosphere of 100% CO_2, 60% CO_2 / 40% N_2, and air combination and then stored at 1°C for 120 days. Physicochemical analyses such as acidity, reduced sugar, bitterness, color (CIE L^*, a^*, b^*) and dry matter contents along with sensory analyses were carried out at regular intervals throughout the storage period. Insignificant differences were determined among washed and unwashed *Hurma* olives in terms of their physicochemical parameters whereas unwashed olive samples retained sensory characteristics better and preferred more by sensory panelists than washed counterparts. Washed and vacuum packaged samples had more undesired values in terms of the sensory characteristics studied.

Keywords: *Hurma* olive, Erkence, Modified atmosphere packaging, De-bittering, Sensory

Hurma Zeytinin (*Erkence cv.*) Fizikokimyasal ve Duyusal Özellikleri Üzerinde Paketleme Öncesi Uygulamaların ve Farklı Gaz Kompozisyonlarının Etkisi

ÖZ

Hurma zeytin, meyve ağaç üzerindeyken acılığını kaybeden özel bir zeytindir. Hasattan sonra doğrudan tüketilebilir. *Hurma* zeytinin tuzsuz olarak muhafazasına yönelik çalışmalar çok sınırlı kalmış ve hayata geçirilememiştir. Bu çalışmada, depolama boyunca *Hurma* zeytinin bazı kalite özellikleri araştırılmış ve farklı gaz kompozisyonları bu zeytinde ilk defa denenmiştir. Bu amaçla 1°C'de 120 gün depolama sırasında yıkama, laktik aside daldırma ve modifiye atmosfer paketlemenin etkinliği incelenmiştir. Zeytinler paketleme öncesinde, çeşme suyu ile yıkanmış ve 1 dakika süreyle %0.2 (w/v)'lik laktik asit çözeltisine daldırılmıştır. Zeytinler vakum uygulamasından sonra %100 CO_2, %60 CO_2 / %40 N_2, ve hava şeklindeki gazlarla paketlendikten sonra 1°C'de 120 gün depolanmıştır. Depolama boyunca asitlik, indirgen şeker, acılık, renk (CIE L^*, a^*, b^*), kuru madde gibi fizikokimyasal analizler ile duyusal analizler yürütülmüştür. Yıkanmış ve yıkanmamış zeytinler arasında fizikokimyasal analizler açısından önemli fark bulunmazken, duyusal özellikler bakımından daha iyi sonuçlar veren yıkanmamış zeytinler panelistler tarafından daha çok tercih edilmiştir. Yıkanmış ve vakumla paketlenmiş zeytinlerin duyusal açıdan daha kötü değerlere sahip olduğu tespit edilmiştir.

Anahtar Kelimeler: *Hurma* zeytin, *Erkence*, Modifiye atmosfer paketleme, Hurmalaşma, Duyusal

INTRODUCTION

Olive is a fruit species that is widely cultivated in Mediterranean countries. Spain, Egypt and Turkey are known to be the leading countries in table olive production [1]. It is not possible to consume olive fruit directly because of oleuropein, which is the most abundant phenolic compound in olive fruit and is responsible for the characteristic bitterness [2]. In order to remove the bitterness of the fruit, a series of processes are necessary. Alkali treatments (sodium or potassium hydroxide) or treatments without alkali such as dry salt and brine are the applications used for the purpose of removing the bitterness in olive. Spanish Style Green Olives, Ripe Black Olives [3] are known to be produced via alkali treatment, whereas brined style Turkish and Greek black olives are the most common examples for alkali-free methods among table olive processing. These methods applied to remove the bitterness in the olive fruits generally require long periods of time, which is undesirable for the producer companies because of economic concerns. Besides, the olives processed via alkali-free treatments contain high amounts of salt as a result of the applied method. Olives belonging to *Erkence* cultivar, which are cultivated in Karaburun peninsula in Turkey, can be directly consumed after harvesting without any further treatment, since fruits are de-bitter on the trees in this cultivar. This naturally de-bittered olives are called as "*Hurma*". Similar varieties were reported to be seen in Greece (*Thrubolea* or *Thoruba Thassos*) [4, 5] and in Tunis (*Dhokar*) [6, 7]. According to the observations of the producers in the region, the climate characteristics of the peninsula (such as wind and dew) constitutes the main effect in the formation of *Hurma* olives. They claim that natural de-bittering needs northern winds and dew. On the other hand, an ancient Greek document mentioned about a fungus, *Phoma olea*, responsible for the formation of *Hurma* olives by breaking down the oleuropein [8]. In addition to the high amount of effective compounds in terms of human health existing in olive tree and products [9], *Hurma* olive is a substantial product for those having hyper tension, since it is not processed with salt treatments and the possible consumption of *Hurma* olives right after harvest can compensate for some of the natural nutrients needed by the human metabolism [10]. Producers only apply salt treatment to *Hurma* olives in case they cannot sell the product in a short period of time after harvest. This case causes *Hurma* olives to lose their unsalted characteristic. Sozbilen and Baysal reported that *Hurma* olives seem safe for human consumption and they recommended some practices such as good handling and preventing contamination strategies to inhibit growth of microorganisms in *Hurma* olive after maturation [11].

MAP has been applied to many food products such as fruits and vegetables in order to extend their postharvest storage, marketing and shelf-life. Oxygen, nitrogen, carbon dioxide and their combinations were reported to be the most common used gases for MAP technique [12, 13, 14]. MAP was also reported to be more effective when applied with low-temperature storage techniques [12, 13]. Before packaging, several techniques such as acetic acid steam treatment, radiation, lactic acid/sodium lactate solution treatment, use of bacteriocin [12], surface disinfection (decontamination) [15] and after packaging gamma irradiation [16] have been carried out in order to increase the efficiency of MAP. In this study, the aim was to determine the physicochemical and sensory changes in unsalted *Hurma* olives, which were treated with surface disinfection and packaged under different gases, during the storage at 1±1°C for 120 days. To our knowledge, this is the first use of MAP for Hurma olives.

MATERIALS and METHODS

Hurma Olives

Since *Hurma* olives fall off the tree spontaneously after de-bittering, the samples were harvested via collecting them from the ground. *Hurma* olives belonging to *Erkence* cultivar, which were cultivated in Guzelbahce/ Izmir (latitude 38.317561° N, longitude 26.924311° E) and fell off at the beginning of December in 2009 after de-bittered on the tree, were used as fruit material.

Processes before Packaging

Olive samples that are damaged, partly de-bittered or not de-bittered were manually separated after harvesting. After separation, the *Hurma* olives that are going to be used for the experiment were divided into two groups. The first group of the samples were packaged without applying any washing process (T_0). The second group was washed with tap water in order to remove the coarse dirt and mud from the sample. Then the fruit samples were immersed in 0.2% lactic acid solution for 1 minute and let to drain for 10-12 hours before packaging (T_1).

Packaging

All the samples weighed into polyamide polyethylene vacuum bags, and each bag contained 250 g of Hurma olives. The properties of the polyamide polyethylene vacuum bags used in the experiment were: thickness=90±3µm, O_2 transmission rate 30 cc/24h/m²/atm, N_2 transmission rate 130 cc/24h/m²/atm, and vapor transmission rate 100 g/24h/m². The samples were packaged using a packaging unit (Baskan Mentel, Sakarya, Turkey) under vacuum, 100% CO_2, 60% CO_2/40% N_2 and normal atmosphere.

Storage

Packed olive samples were stored at 1±1ºC for 120 days in a temperature controlled storage room in Ege University. Physicochemical and sensory measurements were carried out on fruit samples before packaging and 30-day intervals during the storage. Physicochemical analyses were carried out in triplicate, whereas sensory tests were carried out each month as one session.

Physicochemical Analyses

Titratable Acidity

In order to determine the titratable acidity of *Hurma* olives, the samples were homogenized after the removal of pits with Ultra Turrax T25 D blender (IKA®-Ultra Turrax, Germany), and 10 g of homogenized sample mixed with hot distilled water and made up to 250 mL and filtered through filter paper. 10 mL of the filtrate was titrated with 0.1 N NaOH until the pH of 8.1, and results were expressed as m/m% lactic acid according to TS 1125 ISO 750 [17].

Reduced Sugar

According to the Luff-Scroll method, 5 g of pitted and homogenized olive sample was mixed with 5 mL Potassium Ferro cyanide (15%) and 5 mL Zinc sulfate (30%) solutions. Afterwards, the mixture was made up to volume in a 100 mL volumetric flask with distilled water and set aside for a night in a closed sample container. The sample mixture was filtered on the next day and 25 mL Luff solution was added to the filtrated solution. The solution was boiled for 10 min in a heater-condenser. Then, 10 mL Potassium iodate, 25 mL Sulfuric acid (25%) and a few drops of starch solution (5%) was added to the immediately cooled sample solution. The solution was titrated with 0.1 N Sodium thiosulfate solution until the color of the solution turned into creamy yellow. The results were calculated considering the volume of the titration solution consumed for the blank sample and the dilution carried out. Then they were expressed as m/m % [18].

Bitterness

A 50 g sample was taken from pitted and homogenized olives. It was boiled for 5 min after adding 125 mL distilled water and then filtered under vacuum. The residue on the filter paper was washed with 125 mL of distilled water and collected in a beaker. The mixture in the beaker was boiled for 5 min and filtered. The filtrates were collected and then made up to 200 mL with distilled water. 2.5 mL of the filtrate was mixed with 1 mL gelatin (1%) solution. Then, it was made up to 25 mL with acetone and stirred. 20 mL of this solution was poured into 4 g Aluminum oxide, stirred and set aside for 2 min. After the phases were separated, the absorbance of the supernatant was measured with a spectrophotometer (UV-1700 PharmaSpec, Shimadzu Corp., Kyoto, Japan) at 345 nm wavelength [19].

Color

Color measurements (CIE L^*, a^*, b^*) were carried out using Minolta CR-300 Chromameter (Japan). The device was calibrated with the standard white tile. CIE L^*, a^*, b^* values of 20 olives from each package were measured and the mean L* a* b* values used for statistical analysis. L^* represents lightness, whereas a^* represents chromaticity on a green (-) to red (+) axis and b^* states chromaticity on a blue (-) to yellow (+) axis.

Dry Matter

Dry matter was determined by drying 5 g pitted olive samples from each package in an oven at 104°C, until constant weight was obtained. The weight of the samples was measured before and after drying with a precision of 0.0001 g. Percent dry matter was calculated and expressed as percentage of the fresh sample.

Sensory Evaluation

Sensory evaluations were carried out according to the studies of Altug and Elmaci [20] using Numerical Grading Scales with a panel working at the Olive Research Institute (Izmir, Turkey). Sensory properties of the olive samples were evaluated in terms of hardness, taste loss (panel of 7 people) and preference (panel of 18 people). The statements and scores in the hardness scale were as follows: Very soft (1), slightly soft (2), neither soft nor hard (3), slightly hard (4), very hard (5). Besides, the statements and scores in the taste loss scale were as: excessive amount of taste loss (1), too much taste loss (2), moderate amount of taste loss (3), slight amount of taste loss (4), and no taste loss (5). The statements and scores in the preference scale were as follows: dislike (1), like slightly (2), like moderately (3), like (4), like very much (5). The samples were coded randomly with 3 digit numbers and were presented to panelists as whole fruits in white color plates at room temperature.

Statistical Analyses

The sensory properties of olives were evaluated by a panel of seven people, whereas preference evaluation was carried out with a panel of 18 people, which were considered as replications in statistical analysis. Data were subjected to ANOVA according to a split plot design with three replicates (physicochemical properties), considering pretreatments as main plot and storage period as subplot, by SPPS v11 for Windows (SPSS Inc., USA), and significantly different means were separated by Fisher's Least Significant difference (LSD) test.

RESULTS and DISCUSSION

Acidity

An increase was observed in the acidity levels of both washed and unwashed samples (Table 1). Acid formation as a result of carbohydrate breakdown due to microbial growth [21] and chemical reactions might be the reason for the increase in acidity. Washing process and gases used in modified atmosphere packaging were found to have insignificant effect on acidity during storage (P>0.05). The "sour taste" and the other negative statements indicated by the panelists after the 60^{th} day of storage can be associated with the increase in the acidity level of the olive samples. Another study reported that the pH levels of the dry-salted Thassos type olives that was stored with different MAP applications did not change significantly during storage [22].

Bitterness

The bitterness values of *Hurma* olives before storage were determined approximately the same as the bitterness values of ready-to-eat olives processed via different applications to sweeten (the absorbance value for the unwashed olive samples was 0.527) (Table 1). *Hurma* olives could be reported to be acceptable for consumption in terms of bitterness during storage, since their bitterness values were below 1. The bitterness values for the washed and unwashed olive samples were approximately the same before and during storage (Table 1). It was found out that the modified atmosphere gases and washing process had a significant effect on bitterness during storage ($P<0.05$). According to the data obtained, there is a possible risk for bitterness formation after 90 days of storage (Figure 1b). Oleuropein is the compound responsible for bitterness formation in olives. Aktas et al. stated that the total phenolic compounds and oleuropein content of *Hurma* olives (0-1166.89 $mg.kg^{-1}$) during maturation on the tree were lower compared to the amounts in bitter olives (*Erkence*) [8]. They also reported that not only was oleuropein content influenced but also all the phenolic compounds were affected by the de-bittering period. Susamci et al. confirmed the low concentration of phenolic compounds in *Hurma* olives, particularly bitter glucoside oleuropein, in comparison with that of *Erkence* fruit [23]. In this study, the possibility for the hydrolysis of oleuropein was low since *Hurma* olive samples were not in the brine media. For this reason, insignificant difference in the bitterness content was observed until the 120th day of storage. The bitterness value of *Hurma* olive samples during storage were important in terms of regulating it at the level of consumption and at the level that may provide a good source for human health, as well [5].

Reduced Sugar

Reduced sugar content for the unwashed olives was found 0.28% before storage (Table 1). This value is regarded as low for unprocessed raw olives. The low sugar content may be the result of the breakdown of sugar molecules microbiologically or in chemical reactions during de-bittering that occurs while the olive fruit is still on the tree. The reduced sugar contents of the samples during storage were fluctuant but close to the values obtained before storage. The effect of washing process on the reduced sugar content was found significant during storage ($P<0.05$), whereas MAP was reported to have no significant effect ($P>0.05$). However, comparatively lower reduced sugar values were determined for the washed and air packaged olive samples during storage (Figure 1c). It was reported that the concentration of glucose was also lower in *Hurma* than *Erkence* olives [23]. Sugar might be consumed by the microorganisms for fermentation during storage and this might be linked to the "fermented taste" statements indicated for some of the packages in sensory tests. Fernandez-Diez stated that a significant amount of the sugars existing in the olive fruit consisted of reduced sugars. He also reported that these sugars were converted to lactic acid by homofermentative bacteria and to acetic acid or other metabolites in addition to lactic acid by heterofermentative bacteria, thus the acidity increased [24]. The increase in the acidity was a result of this situation.

Color

Hurma olives generally have brown color. As a typical result of de-bittering, olives gain brown color, while some others are black. L^* values demonstrated a decrease in the first 60 days, whereas a^* and b^* values changed around the values obtained before storage (Table 1). L^* values of unwashed samples were found lower. The water remaining on the surface of the samples after washing is considered to contribute to the increase in the L^* values. The color values of *Hurma* olives leastwise consisted of red ($+a^*$) and yellow ($+b^*$) tones during storage. The effect of washing process on L^*, a^*, b^* values was insignificant ($P>0.05$), whilst MAP had a significant effect on a^* values during storage ($P<0.05$). A change in color that might affect the preferences of panelists was not determined (Data not shown).

Dry Matter

The dry matter content of washed samples increased gradually since they lost more water during storage (Table 1). The olive samples packed with 100% CO_2 lost comparatively more water (Figure 1d). Vacuum occurred in package as a result of CO_2 dissolution in oil is considered to be the reason of the increase in water loss creating a pressure on the samples [25, 26]. The effects of washing process and MAP on dry matter during storage was found insignificant ($P>0.05$).

Sensory Evaluation

Sensory evaluations gave the most important results in terms of determining the consumability of *Hurma* olive samples. Before storage, the olive samples were evaluated between "slightly soft" and "neither soft nor hard" with 2.29 points. This softness was observed during storage as well. Panelists indicated that the unwashed samples were harder than the washed ones (Table 2), although the effect of washing process on hardness was insignificant ($P>0.05$). The washed olives that had been packed under vacuum were determined comparatively softer. The pressure occurred due to vacuum in the package might be the reason of the increase in the softness level. Besides, the activity of the oxidative yeasts might have caused softening in the olives [27]. The softening observed in the olive samples generally decreased the acceptability for the consumer. *Hurma* olives specifically have a completely de-bittered taste. Taste differences were determined between the samples since the olives were de-bittered naturally. Some olives had brown color, soft texture and a sour taste, whereas some had black color, hard texture with a wrinkled surface and the taste of that olive was completely de-bittered. Although not for each package or regularly for each month, "sour taste" and other unfamiliar tastes were particularly started to be stated by the panelists after the 60th day of storage. More taste loss was generally observed in the washed and vacuum packaged olive samples (Figure 2b). Low O_2 levels in the packages were reported to contribute to an undesired taste formation [21, 28]. The level of taste loss was higher for the washed samples when compared to the unwashed ones, however the effect of washing process on taste loss was found insignificant (P>0.05). A slight taste loss (4 points) did not affect the consumability of *Hurma* olives, whereas the rankings below 4 points affected the preferences of the panelists negatively. *Hurma* olive samples that were ranked in terms of preference before storage were given a score of 2.67 points. The reason for such a low preference point was a result of the changes occurred in the sensory characteristics of *Hurma* olives due to the climate conditions in that year. The preference points given for washed *Hurma* olive samples were lower than of unwashed samples, however washing process was found to have insignificant effect on preference (P>0.05).

Table 1 Effect of pretreatments (washing and dipping in lactic acid 0.2%) before packaging on physicochemical parameters (means of three replicates±standard deviation) of *Hurma* olives during storage at 1ºC.

Parameters	Days*	0	30**	60	90	120
Acidity (m/m%)	T_0	0.36±0.04	0.62±0.19	0.55±0.19	0.56±0.11	0.73±0.19
	T_1	0.39±0.03	0.64±0.15	0.51±0.16	0.48±0.08	0.74±0.14
Bitterness (Absorbance)	T_0	0.53±0.02	0.63±0.10^a	0.51±0.05	0.52±0.04	0.60±0.17^b
	T_1	0.53±0.05	0.50±0.03^b	0.48±0.04	0.51±0.04	0.78±0.03^a
Reduced Sugar (m/m%)	T_0	0.28±0.07	0.36±0.11	0.39±0.08	0.24±0.08	0.25±0.08
	T_1	0.35±0.04	0.25±0.11	0.37±0.09	0.33±0.18	0.29±0.09
L^* value	T_0	32.37±0.91	31.62±1.09	31.98±1.21	32.32±1.09	33.19±1.15
	T_1	33.16±1.47	32.18±1.14	32.29±0.84	33.06±1.17	34.17±0.70
a^* value	T_0	5.76±0.39	6.86±0.61	6.66±0.68	6.93±0.72	6.94±0.46
	T_1	5.67±0.71	6.62±0.60	6.38±0.32	7.04±0.65	6.73±0.45
b^* value	T_0	6.47±0.93	8.19±1.12	6.22±0.91	8.79±1.11	8.94±0.85
	T_1	6.81±1.63	7.87±1.27	5.57±0.93	8.61±1.32	8.31±0.84
Dry Matter (m/m%)	T_0	56.41±1.59	56.11±3.31	55.78±2.95	55.48±5.40	57.22±2.85
	T_1	51.31±2.06	55.26±4.36	53.70±4.67	56.19±2.75	55.91±3.34

*T_0, without applying any washing process; T_1, washed+diped lactic acid 0.2%. **Different superscript letters following the values in same column indicate differences during storage period for each parameter (P<0.05)

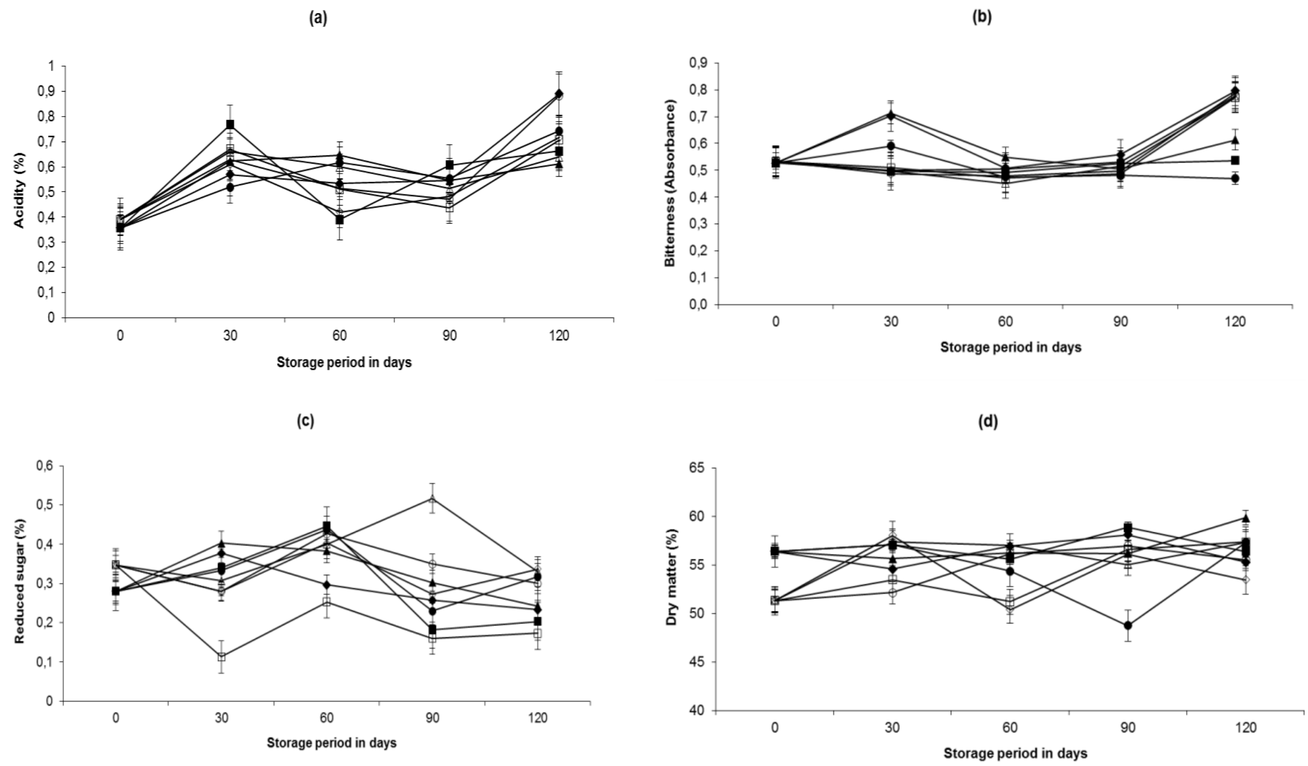

Figure 1. Effect of pretreatments before packaging in combination with MAP on (a) acidity, (b) bitterness, (c) reduced sugar and (d) dry matter of *Hurma* olives during storage at 1ºC. (♦), T_0+vacuum; (◊), T_1+vacuum; (▲), T_0+100%CO_2; (∆), T_1+100%CO_2; (●), T_0+60%CO_2/40%N_2; (○), T_1+60%CO_2/40%N_2; (■), T_0+air; (□), T_1+air. Standard error is shown on the bars.

The points for taste loss and preference were changed correspondingly during storage. Preference points given for washed and vacuum packaged olives were lower when compared to the samples in other packages, since taste loss was higher for washed ones in vacuum packages (Figure 2c). The high loss of taste and the low preference obtained for the washed *Hurma* olives might be regarded as a disadvantage of the surface decontamination process applied before packaging [15].

Table 2 Effect of pretreatments (washing and dipping in lactic acid 0.2%) before packaging on sensory attributes of *Hurma* olives during storage at 1ºC.

Sensorial attribute	Days	0	30	60	90	120
Hardness	T_0	2.29±0.90	2.96±1.00	2.82±0.82	2.46±0.74	2.86±0.85
	T_1	2.29±0.90	2.29±0.81	2.21±0.92	2.43±0.63	2.46±0.79
Taste loss	T_0	4.14±0.85	3.89±1.17	4.11±1.03	4.00±0.90	3.75±0.80
	T_1	4.14±0.85	3.50±1.40	3.75±1.08	3.75±1.08	3.32±0.98
Preference	T_0	2.67±0.95	2.75±0.96	2.83±0.99	2.83±1.02	2.39±1.01
	T_1	2.67±0.95	2.39±0.99	2.44±1.05	2.61±0.86	2.14±0.89

T_0, without applying any washing process; T_1, washed+diped lactic acid 0.2%.

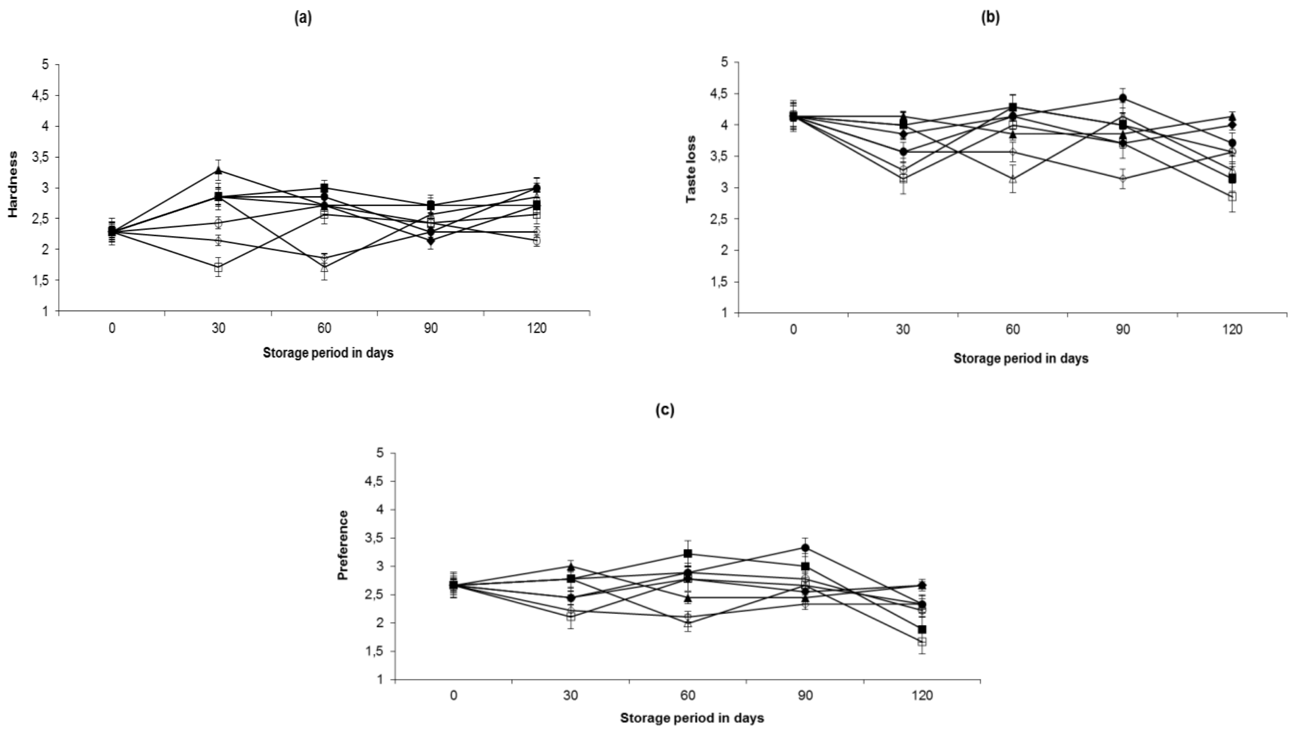

Figure 2. Effect of pretreatments before packaging in combination with MAP on (a) hardness, (b) taste loss and (c) preference of *Hurma* olives during storage at 1ºC. (♦), T_0+vacuum; (◊), T_1+vacuum; (▲), T_0+100%CO_2; (∆), T_1+100%CO_2; (●), T_0+60%CO_2/40%N_2; (○), T_1+60%CO_2/40%N_2; (■), T_0+air; (□), T_1+air. Standard error is shown on the bars.

CONCLUSIONS

The effect of washing process, lactic acid treatment (0.2% w/v) and MAP (vacuum, 100% CO_2, 60% CO_2/40% N_2, air) on the physicochemical and sensory properties of *Hurma* olives was studied. Washing and dipping into lactic acid solution did not cause a significant change during storage in the physicochemical properties of samples, except for bitterness and reduced sugar contents. The sensory properties of *Hurma* olive samples that were washed and dipped into lactic acid solutions were poorer; in particular, vacuum packaged samples among this group were found to have more undesired values in terms of sensory characteristics. The data obtained in this study might provide the basics for marketing *Hurma* olives that were packed with MAP and stored at 1°C for 120 days.

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