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Use of Activated Carbon inside Modified Atmosphere Packages To Maintain Tomato Fruit Quality during Cold Storage

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Ethylene triggers the ripening process of tomato affecting the storage durability and shelf life (loss of quality) and inducing fruit decay. In this paper, an active packaging has been developed on the basis of the combination of modified atmosphere packaging (MAP) and the addition of granular-activated carbon (GAC) alone or impregnated with palladium as a catalyst (GAC−Pd). A steady-state atmosphere was 4 and 10 kPa for O_2 and CO_2 in control packages, while it was 8 and 7 kPa for O_2 and CO_2 in treated ones. The addition of GAC−Pd led to the lower ethylene accumulation inside packages, while the higher was obtained in controls. The parameters related to ripening showed that treated tomatoes exhibited a reduction in color evolution, softening, and weight loss, especially for GAC−Pd treatment. Moreover, these treatments were also effective in delaying tomato decay. After sensorial panel, tomatoes treated with GAC−Pd received the higher scores in terms of sweetness, firmness, juiciness, color, odor, and flavor. Results from the GC−MS analysis of the MAP headspace showed that 23 volatile compounds were identified in control packages, with these volatiles being significantly reduced in MAP-treated packages, which was correlated to the odor intensity detected by panelists after bag opening.

KEYWORDS: **Active packaging; volatile compounds; decay; ethylene adsorber; MAP**

INTRODUCTION

Tomato is considered a climacteric fruit in which ripening is accompanied by a peak in respiration and a concomitant sharp increase in ethylene production, which accelerates quality loss through the physicochemical changes related to this process, such as softening, color evolution, aroma development, among others (*1*, *2*). In addition, ethylene induces an increase of fruit pathogen susceptibility and physiological disorders, with a net reduction in postharvest life. These effects depend upon a number of variables, with the most important being tissue sensitivity to ethylene, duration of exposure, ethylene concentration, atmospheric composition, and temperature (*3*).

Several means have been used to prolong fruit storability during postharvest, such as cold storage, alone or in combination with modified atmosphere packaging (MAP). The choice of packaging system/method is a key factor to obtain optimum modifications of the atmosphere and to avoid extremely low levels of O_2 and/or high levels of CO_2, which could induce anaerobic metabolism with the possibility of off-flavor generation and/or the risk of anaerobic microorganism proliferation (*4*, *5*). The use of MAP in tomato has been found to be effective on the maintenance of quality for either whole fruit (*6*) or fresh-cut slices (*7*, *8*), although ethylene started to accumulate after a few days of storage.

Currently, ethylene biosynthesis and action can be blocked by chemical compounds that differ in their structure and act at different levels, such as modifying ACS (ACC synthase) and ACO (ACC oxidase) activities (*9*−*11*), blocking receptor sites (*12*−*13*), and diversion of SAM through polyamine biosynthesis (*14*−*16*). However, there are many situations in which considerable ethylene emission occurs along the food chain, such as inside the packages, storage chambers, during transportation, and in domestic refrigerators. Then, additional tools to eliminate this ethylene are therefore necessary, as could be the use of activated carbon (*17*). Thus, the aim of this paper has been to study the effect of granular-activated carbon (GAC), alone or in combination with palladium (GAC−Pd), inside MAP packages on maintaining tomato fruit quality during cold storage.

MATERIALS AND METHODS

Plant Material and Experimental Design. Tomato fruits (*Lycopersicon esculentum* Mill. cv. "Beef") were harvested at the turning stage of ripening from a commercial farm in Mazarrón (Murcia, Spain). At laboratory, 1000 tomato fruits were selected to obtain homogeneous batches of four fruits based on color, size, and absence of defects. A total of 10 lots ($n = 40$) were used to determine the fruit characteristics at harvest. The remaining lots were packed in 20 μm thickness nonperforated oriented polypropylene (N-OPP) bags (30 × 20 cm),

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which had permeabilities at 1 °C of 1600 mL of O_2 m^{-2} d^{-1} atm^{-1} and 3600 mL of CO_2 m^{-2} d^{-1} atm^{-1}. This film was selected on the basis of the results of previous experiments (unpublished data).

Activated Carbon Preparation and Treatments. GAC (20−60 mesh with a specific area of 626 m^2 g^{-1}) was purchased from Sigma (Sigma−Aldrich, Madrid, Spain). The GAC was impregnated through the reaction with palladium acetate to obtain a concentration of 1% (GAC−Pd). The following treatments were performed: (a) sachets with 5 g of GAC, (b) sachets with 5 g of GAC−Pd, and (c) control. The sachets were deposited inside the packages just before the sealing of the bags. All packages were stored at 8 °C and with a relative humidity (RH) of 90% in darkness, and weekly 10 bags for each treatment were sampled, in which the following analytical determinations were performed. The remaining bags (40 for each treatment) were used for decay evaluation throughout storage.

Gas Composition. A silicone septum was provided on the bag surface for sampling gas inside the package, in which CO_2, O_2, and ethylene concentrations were quantified as previously indicated (*18*). Results were the mean ± standard error (SE) of two determinations for each of the 10 replicates ($n = 20$).

GC−MS Volatile Analysis. After 14 days of storage, 1 mL of the headspace atmosphere was withdrawn using a gas syringe and injected into a Shimadzu GC−MS QP5050A (Shimadzu, Tokyo, Japan) in splitless. The injector temperature was 250 °C, and the column was Supelcowax-10 (30 m × 0.25 mm, i.d. of 0.25 μm) at a flow rate of 1.7 mL of helium min^{-1}. The temperature program was initial temperature at 50 °C increasing up 240 °C at a rate of 5 °C min^{-1} and a total chromatogram time of 41 min with a holding time of 3 min. The scan mode was used to detect all of the compounds in the range of m/z 45−350. The peaks were identified with a mass spectrometer coupled to the GC by comparison of experimental spectra with those of the NIST (National Institute for Standards and Technology, Gaithersburg, MD) and Wiley data banks or with authenticated standards (octanal, nonanal, and dodecane). The retention index was calculated using a mixture (C8−C32) from Sigma (Sigma−Aldrich, Madrid, Spain). Results were the mean ± SE of two determinations for each of the 10 replicates ($n = 20$) and expressed as the peak area.

Weight Loss. The weight of individual bags was recorded on the day of harvesting and after the different sampling dates. Cumulative weight losses were expressed as the percentage loss of the original weight and were the mean ± SE of 10 bags.

Color. Skin and mesocarp color were determined using the Hunter Lab System and a Minolta colorimeter CR200 model (Minolta Camera Co., Osaka, Japan). Color was expressed as a* parameter, and results were the mean ± SE of determinations made in duplicate on each fruit for each bag along the equatorial axis ($n = 80$).

Firmness and Maturity Index Determination. Fruit firmness (N mm^{-1}) was determined using a flat steel plate mounted on a TX-XT2i Texture Analyzer (Stable Microsystems, Godalming, U.K.) as previously reported (*18*). The maturity index was expressed as the ratio between the total soluble solids concentration (TSS) and titratable acidity (TA). For each bag, a segment of each fruit was taken and homogenized in which measurements of TSS and TA in duplicate were performed (*18*).

Decay Analysis. For decay analysis, the number of bags with the presence of at least one decayed tomato was monitored through storage and then the decay percentage from the total bags was calculated for each treatment and sampling date.

Sensory Evaluation. Sensory analyses to compare the internal package odor of treated and control tomatoes were carried out by 10 trained adults, aged 25−40 years (5 females and 5 males). The panel was trained in a pretest for tomato aroma from whole fruits. A laboratory of sensory analyses with an individual booth for each panelist was used. For each treatment and sampling date, the 10 bags were opened and each judge evaluated immediately the off-flavor odor intensity on a ranked scale of 1 to 5, where 1 = absence, 2 = slight odor, 3 = moderate, 4 = severe off-flavor, and 5 = extremely off-flavor ($n = 10$). After 14 days of MAP storage, the same panel evaluated the following tomato quality attributes: color, firmness, odor, juiciness, turgidity, sweetness, sourness, and flavor, on a descriptive analysis using a 10-cm unstructured line scale to evaluate the descriptors. Panelists were pretrained in visualization, smelling, and tasting tomatoes (either whole or cut). Each panelist evaluated two tomatoes from each treatment, and results were the mean ± SE ($n = 20$).

Statistical Analysis. Data for the physical, chemical, decay, and sensory parameters were subjected to analysis of variance (ANOVA). Sources of variation were the time of storage and treatments. Mean comparisons were performed using HSD Tukey's test to examine if differences between treatments and storage time were significant at $p < 0.05$. All analyses were performed with SPSS software package version 11.0 for Windows.

RESULTS

Gas Composition Inside the Packages. A sharp decrease in O_2 and a concomitant increase of CO_2 was observed in MAP packages reaching the steady-state atmosphere after 7 days of cold storage with levels of ≈4 kPa O_2 and ≈10 kPa CO_2 in controls (**Figure 1**). However, in MAP packages with GAC or GAC−Pd, significantly lower concentrations of CO_2 and higher of O_2 were obtained without significant differences between them (≈7 kPa O_2 and ≈7 kPa CO_2). Control packages exhibited a drastic increase of ethylene after 1 day of storage (48.16 ± 4.87 μL L^{-1}) followed by a continuous diminution. Contrarily, ethylene was significantly lower in MAP packages with GAC and especially in those with GAC−Pd (**Figure 1**).

Parameters Related to Tomato Quality. Weight loss was very low but control tomatoes showed the highest weight loss at the end of the experiment (0.71 ± 0.06%), while tomatoes with GAC or GAC−Pd (**Figure 2**) exhibited significantly lower weight losses (0.54 ± 0.02 and 0.49 ± 0.03%, respectively). The measurement of color revealed that the a* parameter showed the greater changes by increases during storage for both skin and mesocarp tissues (**Figure 3**). However, these changes were more pronounced in control tomatoes with values of 6.35 ± 1.21 and 8.02 ± 031 for skin and mesocarp color after 28 days of storage, respectively. At this time, tomatoes with GAG or GAC−Pd reached values of ≈2 (skin) and ≈7 (mesocarp) without significant differences between them. Fruit firmness at harvest (4.76 ± 0.15 N mm^{-1}) decreased significantly during storage, although the loss of firmness was higher in control fruits than in those with GAC or GAC−Pd, especially during the first 14 days of storage (**Figure 4**). TSS and TA at harvest were 6.93 ± 0.07 °Brix and 0.93 ± 0.02 g 100 g^{-1} equiv of citric acid, respectively, rending a maturity index (TSS/TA ratio) of 7.45 ± 0.08. During storage, a diminution of TA (ca. 0.60 g 100 g^{-1} equiv of citric acid) and maintenance of TSS were observed with no significant differences with or without the addition of GAC (data not shown).

Decay Incidence. The occurrence of decay in control tomato started after 21 days of cold storage, at which 40% of the bags showed some fruit with fungal growth. This percentage reached 100% after 28 days (**Figure 5**). Contrarily, the presence of the decay was delayed on time in tomatoes with GAC or GAC−Pd, for which any decayed tomato was detected after 21 days. Moreover, the occurrence of decay after 28 days was significantly lower (39%) for GAC−Pd than tomatoes with GAC (65%).

Sensory Evaluation. The intensity of odor was evaluated by judges after immediately opening the bags and was judged as severe off-flavor occurrence (scores ≈ 4) in control packages from the first sampling date (**Figure 6**). On the contrary, no off-flavor was detected in those packages with GAC and especially with GAC−Pd, in which the scores were <2. These results were confirmed to those obtained from the headspace volatile compounds analysis by GC−MS, which revealed that there were significant differences among packages. In control

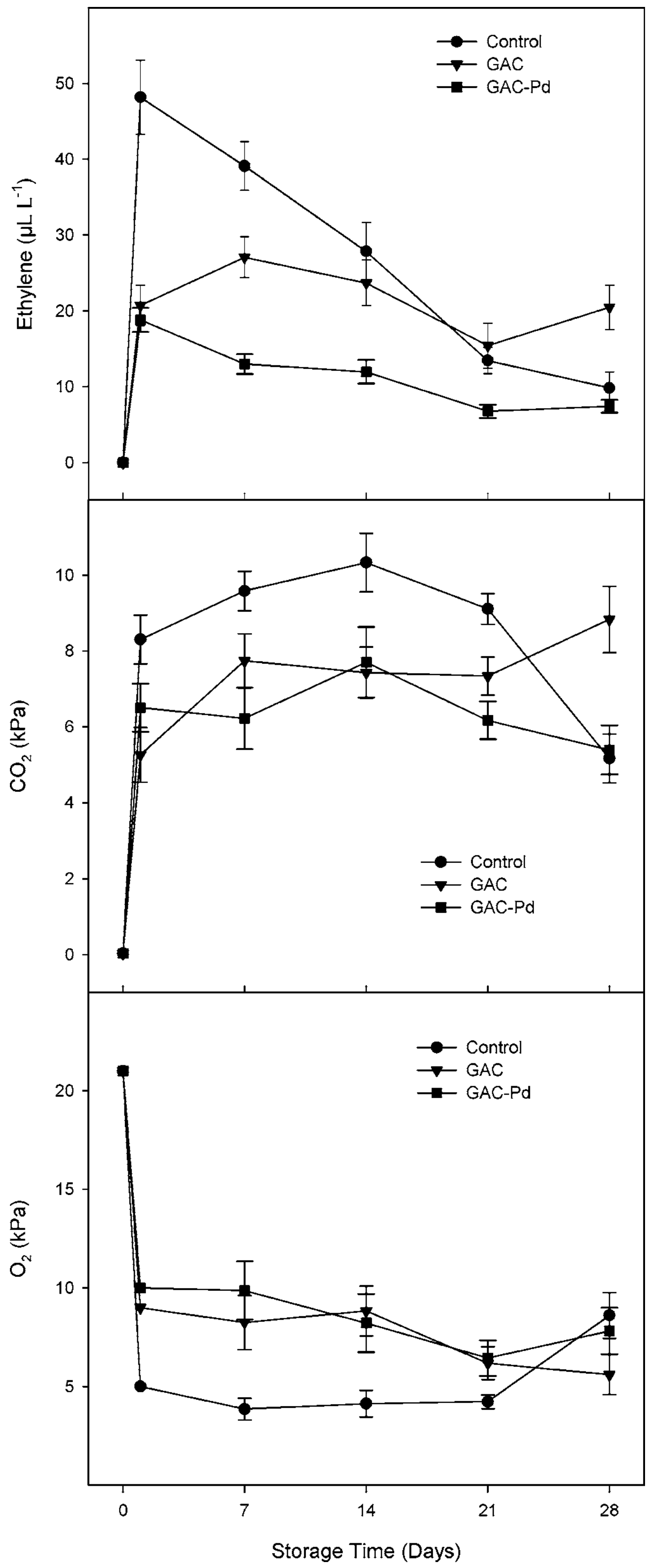

Figure 1. Ethylene, CO_2, and O_2 concentrations inside MAP packages along storage.

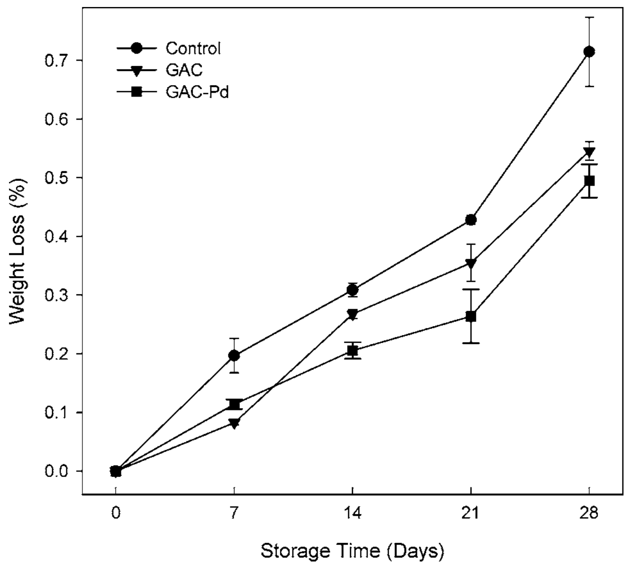

Figure 2. Percentage of weight loss throughout storage in MAP-packaged tomatoes.

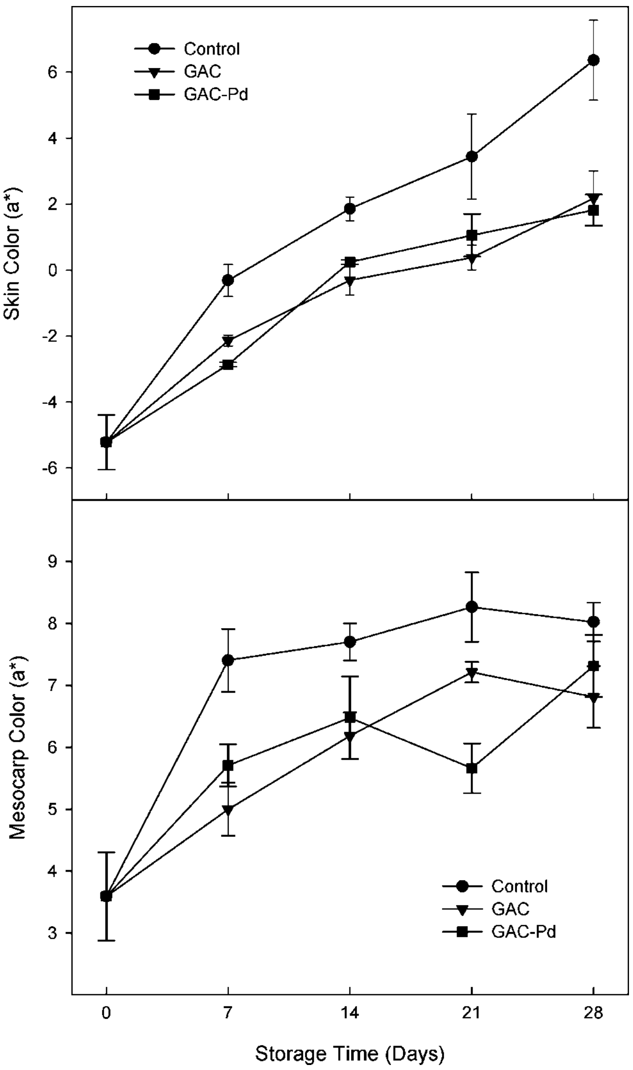

Figure 3. Evolution of skin and mesocarp color (a* parameter) throughout storage in MAP-packaged tomatoes.

bags 23 peaks were identified (**Table 1**), from which the main functional groups were aldehydes (55%) followed by alcohols (17%), hydrocarbons (15%), esters and ketones (11%), and acids with a very low percentage (1%). However, in the bags with GAC or GAC−Pd, six of these compounds also appeared although with peak areas significantly much lower. Thereafter, two volatile compounds (peak numbers 4 and 8) occurred in bags with GAC or GAC−Pd only. Moreover, in both cases, the main compounds were alcohols (≈55%) followed by aldehydes (≈35%) and esters (≈10%), but the absence of ketones, hydrocarbons, and acids were observed. Panelists

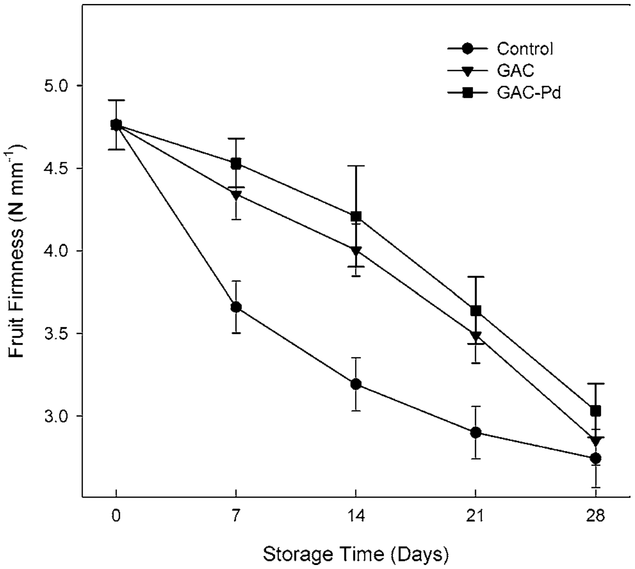

Figure 4. Changes in fruit firmness throughout storage time in MAP-packaged tomatoes.

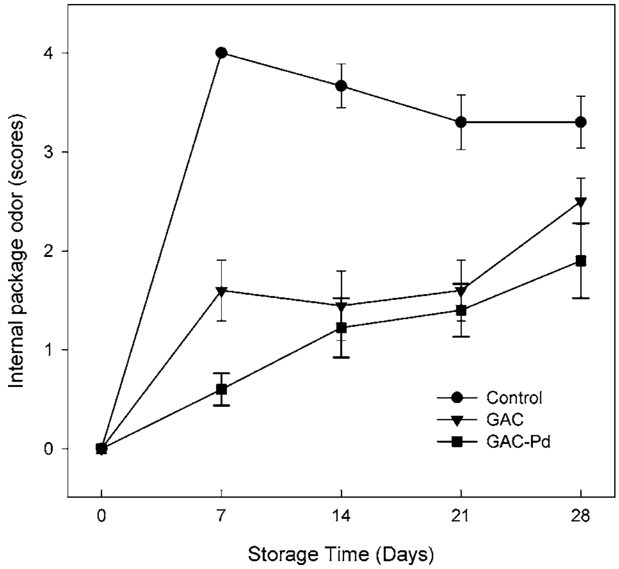

Figure 6. Scores for internal package odor after immediately opening the bags throughout storage time in MAP-packaged tomatoes.

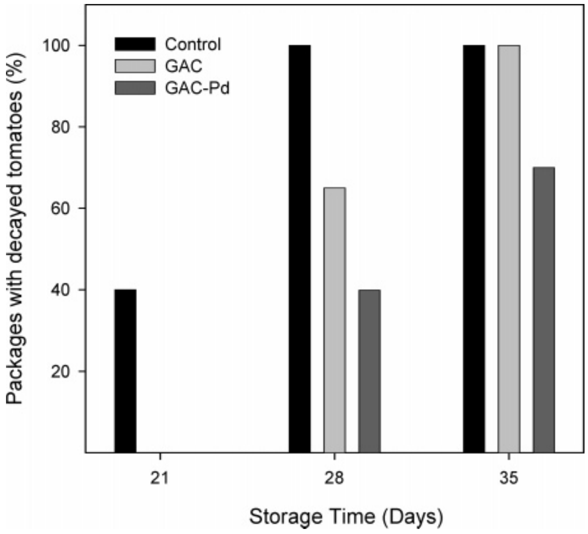

Figure 5. Percentage of bags that showed some decayed fruit throughout storage time in MAP-packaged tomatoes.

evaluated the quality of tomatoes after 14 days of storage and gave the highest scores for sweetness, juiciness, odor, and flavor in tomatoes from bags with GAC or GAC−Pd (**Figure 7**), while no significant differences were found for sourness and turgidity between control and treated tomatoes. The attributes with most significant differences were color and firmness, for which the GAC−Pd had the highest scores.

DISCUSSION

In this paper, MAP has been combined with ethylene adsorbers such as GAC alone or with palladium as a catalyst (GAC−Pd) as a mean of the preservation of tomato quality during postharvest storage. In fact, the addition of GAC and especially GAC−Pd reduced efficiently the ethylene accumulation inside MAP packages, according to previous reports in fruits and vegetables (*19*, *20*). During storage, the ethylene concentration decreased inside the packages, which could be attributed to the effect of high CO_2 and low O_2 concentrations on inhibiting the ethylene production rate (*8*), especially in controls. Moreover, the steady-state atmosphere that was reached could be considered as optimum to extend the shelf life of tomato according to previous reports on whole and sliced tomatoes (*8*, *21*).

The parameters related to ripening, such as changes in color, firmness, and weight loss evolved more slowly in tomato from packages with GAC or GAC−Pd. Because these parameters are known to be triggered and regulated by ethylene production in tomato, as well as other climacteric fruits (*2*), the addition of an adsorbent inside the packages led to a delay of the tomato ripening process and, in turn, an extension of the storability period. In addition, the MAP itself has an effect on delaying the color development of tomato fruit when levels of CO_2 higher than 9 kPa and/or O_2 levels lower than 12 kPa are achieved (*22*, *23*). The texture is an important attribute demanded by consumers and most of the time responsible for fruit acceptability and postharvest shelf life. In fact, the addition of GAC or GAC−Pd significantly reduced the firmness losses during cold storage, which could be attributed to the lower ethylene accumulation, because tomato softening is sensitive to ethylene (*24*). Moreover, these treatments were effective in reducing the percentage of tomato decay, which is mainly due to species of *Penicillium* and *Botrytis* (*8*, *25*).

The internal package odor was highly scored in controls compared to those obtained in bags with GAC or GAC−Pd. These differences were confirmed by the occurrence of volatiles, which were increased in the controls. In addition, the generation of off-flavor in control bags could be related to the high accumulation of ethylene inside the packages, as has been shown to occur in other fruits (*26*). A large number of volatile compounds (over 400) have been identified in fresh and processed tomato products (*27*), although 20−30 have proven to be the most important compounds contributing to the aroma. There are only a few papers on tomato storage under MAP conditions and the changes in aroma compounds (*28*, *29*), but no available references have been found on the volatile composition in the headspace atmosphere inside the bags, because the majority of determinations have been performed in blended tissue.

In this paper, after 14 days of storage, 23 compounds were identified in control MAP, with the main ones being aldehydes and alcohols, as has been reported in other tomato cultivars (*29*−*34*). Among the volatile compounds, nonanal was the predomi-

Table 1. Volatile Compounds from the Headspace of MAP Package Tomatoes after 14 Days of Cold Storage

peak number	retention index	volatile compound	control area	relative percentage	GAC area	relative percentage	GAC–Pd area	relative percentage
1	700	heptane	810 087	14.12				
2	848	2-methylbutan-1-ol	668 331	11.65	582 566	42.62	307 575	40.32
3	928	4-methylhexanal	606 825	10.58				
4	932	pentyl formate			162 442	11.88	90 828	11.91
5	988	heptan-1-ol	133 278	2.32				
6	1002	3-methylpentanoic acid	25 228	0.44				
7	1017	octanal[a]	657 295	11.46				
8	1020	3-methylpentan-1-ol			171 289	12.53	95 172	12.48
9	1070	dodecan-1-ol	57 816	1.01				
10	1084	octan-1-ol	108 110	1.88				
11	1090	butanoic acid	23 579	0.41				
12	1113	nonanal[a]	864 061	15.06	229 223	16.77	121 662	15.95
13	1116	cyclopentanone	26 896	0.47				
14	1166	non-2-enal	123 795	2.16	31 569	2.31	15 307	2.01
15	1180	pentanoic acid	10 830	0.19				
16	1197	5-methylhexan-2-one	26 726	0.47				
17	1200	dodecane[a]	29 461	0.51				
18	1213	decanal	127 366	2.22	32 877	2.41	15 149	1.99
19	1268	dec-2-enal	455 416	7.94	96 688	7.07	71 188	9.33
20	1277	acetic acid	1 111	0.02				
21	1307	dodecane-1,1-difluoro	22 873	0.40				
22	1313	dodecanal	33 868	0.59				
23	1360	2,2,4-trimethyl-1,3-pentanedioldiisobutyrate	237 022	4.13				
24	1370	undec-2-enal	299 797	5.23	60 274	4.41	45 993	6.03
25	1381	(3-hydroxy-2,4,4-trimethyl-pentyl) 2-methylpropanoate	386 865	6.74				
		total area	5 736 635	100	1 366 927	100	762 873	100

[a] Authenticated standards. The remaining peaks are tentative.

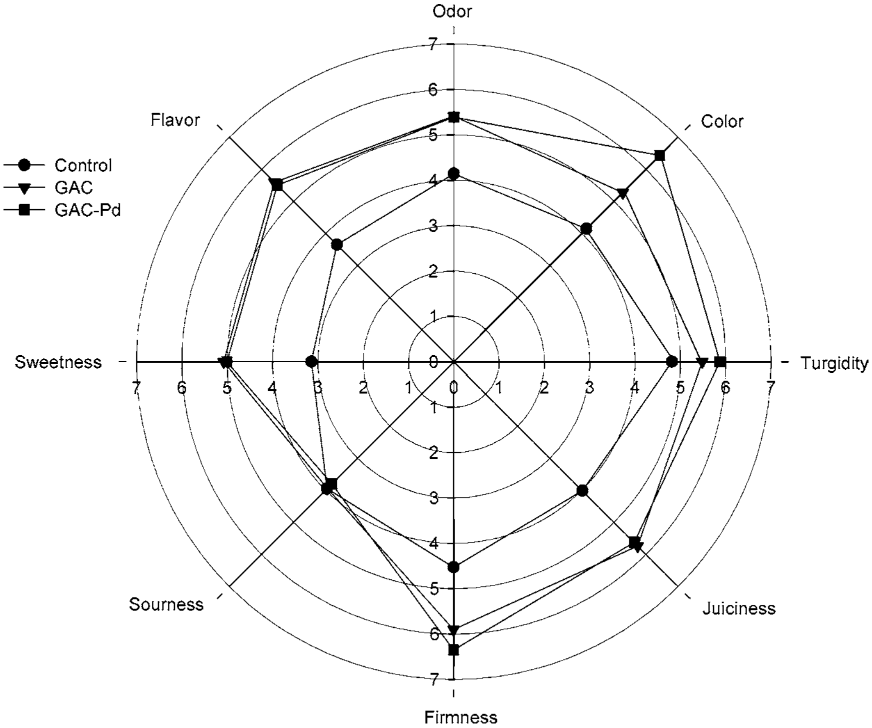

Figure 7. Results from the sensorial analysis for tomato-quality parameters after 14 days of storage.

nant (15%) followed by heptane (14%), 2-methylbutan-1-ol (12%), 4-methylhexanal and octanal (11%), and dec-2-enal (8%). This profile slightly differed from that obtained in macerated tomatoes, mainly because of the effect of tissue disruption through the generation of C6 compounds, such as hexanal, *cis*-3-hexenal, *trans*-2-hexenal, *cis*-3-hexenol, and hexenol, among others (*32*, *35*, *36*). Thus, hydrogenated hydrocarbons (heptane and dodecane) have been detected in the headspace of nonmacerated tomatoes, while these compounds were not identified in previous reports of blended tomatoes. When GAC or GAC−Pd were added inside the packages, a net decrease of volatile compounds occurred, because just eight compounds were identified with peak areas significantly lower than in control bags. In treated packages, the alcohols became the predominant while acids, ketones, and hydrocarbons disappeared. Accordingly, the addition of ethylene adsorbers led to a decrease of volatiles in MAP-packaged bananas (*37*). It is interesting to point out that this decrease of volatiles was not correlated to those results obtained from the sensorial panel, because panelists judged better the quality attributes of tomatoes with GAC or GAC−Pd than controls in terms of odor and flavor. One possible explanation could be due to the high percentage of 2-methylbutanol (over 40%) of the total volatiles. This compound derived from the amino acid leucine (*35*) and has been described as one of the main components contributing to the sweet/fresh ripe tomato flavor (*31*, *38*, *39*). Moreover, other attributes related to the tomato quality such as juiciness, color, sweetness, and firmness also received higher scores in treated rather than in control tomatoes. The visual aspect of tomatoes after 21 days of MAP storage, either whole or cut, clearly showed the beneficial effect of the addition of GAC or GAC−Pd to MAP packages in term of delaying the ripening process and the occurrence of decay. Thus, control tomatoes were redder and darker and showed a higher incidence of fungal growth than those from MAP-treated packages.

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