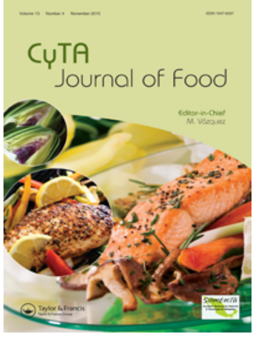

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# Effects of packaging and storage conditions on quality and volatile compounds of raspberry fruits

# Los efectos de las condiciones de embalaje y almacenamiento en la calidad y los compuestos volátiles de la frambuesa

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Modified atmosphere packaging (MAP) has been found to extend the shelf life of raspberries, but temperature fluctuations could result in quality and aroma changes. Fruits cv. Himbo Top were evaluated after wrapping with biodegradable and polypropylene (PP) films under passive and active MAP conditions for 96 h (48 h at 1°C followed by 48 h at 18°C). A PP macro-perforated film was used as control. After 48 h the biodegradable film maintained fruit colour parameters near to harvest value (L 26.9 and 27.6, respectively for passive and active atmospheres) and, as the temperature increased, it was the only film used that facilitated storage of fruit for up to 96 h (24.4–25.9 kPa of $CO_2$). Qualitative and quantitative differences were found in the initial 54 volatile compounds; after 96 h raspberries stored under passive and active MAP showed a similar aroma profile, mainly dominated by terpenes (73 and 62%, respectively).

**Keywords:** aroma compounds; modified atmosphere; film; temperature; quality

Se ha encontrado que el embalaje en atmosfera modificada (MAP) extiende el periodo de conservación de las frambuesas, aunque las fluctuaciones de temperatura podrían resultar en cambios en la calidad y el aroma. Se evaluaron las frutas cv. Himbo Top después de embalar con papeles film biodegradables y de polipropileno bajo condiciones MAP pasivas y activas durante 96 horas (48 horas a 1°C seguido de 48 horas a 18°C). Se utilizó un papel film macroperforado de polipropileno como control. Después de 48 horas el papel film biodegradable mantuvo los parámetros de color de la fruta cerca del valor añadido (L 26,9 y 27,6 respectivamente en la atmosfera pasiva y activa) y a medida que aumentaba la temperatura fue el único papel film capaz de almacenar frutas un máximo de hasta 96 horas (24,4–25,9 kPa de $CO_2$). Se encontraron diferencias cualitativas y cuantitativas en los 54 compuestos volátiles iniciales; después de 96 horas las frambuesas almacenadas bajo MAP pasivas y activas mostraron un perfil aromático similar, sobretodo predominado por terpenos, 73% y 62% (respectivamente).

**Palabras clave:** compuestos aromáticos; atmosfera modificada; papel film; temperatura; calidad

## Introduction

Raspberry fruits (*Rubus ideus* L.) are a highly perishable product and are best stored at low temperature and a high relative humidity (RH), ideally 0.5°C and >95% (Haffner, Rosenfeld, Skrede & Wang, 2002; Krüger, Dietrich, Schöpplein, Rasim & Kürbel, 2011). It can be difficult to maintain such a low temperature during postharvest handling, and consequently modified atmosphere packaging (MAP) has been used to extend the shelf life of these fruits. When raspberries are stored in gaseous mixtures containing 10% $O_2$ and 15% $CO_2$, decay is significantly reduced (Siro, Devlieghere, Jacxsens, Uyttendaele & Debevere, 2006) and the fruits show a more attractive colour compared with those stored under normal atmosphere (NA) conditions (Haffner et al., 2002). Reduced $O_2$ levels and elevated $CO_2$ levels have been shown to reduce the respiration rate of fruits (Beaudry, 1999), but the temperature fluctuations that can occur during storage, transport and retail display can generate an unfavourable atmosphere inside the package (Nunes, Emond, Rauth, Dea & Chau, 2009) and the loss of aroma compounds. High $CO_2$ concentrations can disrupt enzyme systems, such as the lipoxygenase pathway which is involved in the formation of aromatic volatile compounds, while total ester content – and particularly methyl dihydrojasmonate – may decrease after cold storage (Morales et al., 2014). The hydrophobicity and size of aroma compounds as well as the crystallinity of the wrapping film are critical factors that can affect the adsorption and transfer of volatile components in packaged products (Cava, Lagaron, Lopez-Rubio, Catala & Gavara, 2004). Interactions between traditional packaging made from petroleum-based polymers and the flavour consituents can cause adsorption and absorption of flavour volatiles by the packaging material; for example, the permeation of flavour volatiles through the plastic material, food- and flavour-induced changes in the physical properties of the plastic polymer, as well as the interaction of low-molecular weight compounds in the plastic – such as solvent and plastifiers – with the food flavour or the food itself, can all result in an overall imbalance in the flavour profile of the food (Sajilata, Savitha, Singhal & Kanetkar, 2007). In some cases volatile aroma components interact with the polymer matrix, lowering their concentration in the package headspace; petroleum-based polymers, such as polypropylene (PP), can absorb a wide variety of flavour compounds including esters, ketones and aldehydes (Caner, 2011). To provide an adequate shelf life and product quality, packaging materials must preserve the overall flavour management of the packaged raspberries; the loss of aroma does not alter the product in terms of

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*Corresponding author. Email: nicole.giuggioli@unito.it its nutritional, microbiological or toxicological properties, but it affects the perception of food quality by the consumer (Caner, 2011). The volatile aroma profile of raspberries in fact represents an important factor in consumer acceptability and is associated with other factors, such as their sweet taste related to correct sugar/acid balance, external colour derived from anthocyanins and their health-giving properties. Different packaging solutions have been evaluated in regard to extending the shelf life of raspberries (Giovanelli, Limbo & Buratti, 2014; Peano, Girgenti, Palma, Fontanella & Giuggioli, 2013; Seglina et al., 2010), but limited data are reported on the influence of both novel materials used for MAP and the effect of varying storage temperature in the supply chain on the content of volatile compounds in fresh raspberries. Research on starch-based films has shown that such films may be a suitable alternative to conventional plastics for various food products (Peano, Girgenti & Giuggioli, 2014; Peelman et al., 2013). The aim of the present work was to evaluate the most important qualitative traits and volatile compounds of red raspberry variety cv. Himbo Top after wrapping the fruits in a non-commercial biodegradable compostable film and a PP film under passive and active MAP conditions for 96 h (48 h at 1°C followed by 48 h at 18°C).

## Materials and methods

### *Fruit samples*

Raspberry cv. Himbo Top was obtained from a commercial orchard (Agrifrutta Soc. Coop. S.R.L., Piedmont, Italy). The fruits were picked by hand at the end of July at the red-ripe stage of maturity, individually deposited in polyethylene terephthalate (PET) trays and immediately transferred to the laboratory under cold conditions (5 ± 1°C and 80–85% RH). Damaged and mouldy fruits were eliminated (by hand) prior to analysis. The various storage treatments were started approximately 3 h after harvest.

### *Packaging and storage conditions*

For the trials, raspberries were placed in PET trays (0.150 kg per tray) and wrapped with three different single-layer films under different gas compositions.

The films used were: (1) non-commercial biodegradable and compostable film; (2) non-perforated prototypes (Novamont, Italy); (3) non-perforated commercial PP film (Trepack, Italy); and (4) commercial PP macro-perforated film (6 mm holes; Trepack, Italy), the one used in the retail business. In regard to flow pack equipment, an electronic horizontal wrapping machine (Taurus 800, Delphin, Italy) was used, including a take-up reel with translational movement of the jaws. Gas addition was conducted using a PBI Dansensor (Italy) gas mixer that allowed for different gas mixtures ($O_2$, $CO_2$, $N_2$) to be used for each packed tray. The films, treatments (different packaging materials), permeability properties and relative modification of the atmosphere inside each tray are reported in Table 1. All fruits were stored at 1 ± 1°C in a cold room held at 90–95% RH for 48 h in accordance with the storage procedures of the packing house. After cold storage, the fruits were held for an additional 48 h at 18 ± 1°C to simulate retailer conditions.

### *Sampling procedures*

All analyses, with the exception of headspace gas composition (daily sampling), were performed for each treatment at harvest (0) and at the end of storage, both at 1 ± 1°C (48 h) then at 18 ± 1°C (96 h). Three randomly selected trays (0.375 kg of raspberry fruits) were used for each treatment.

### *Headspace gas composition*

Carbon dioxide and oxygen concentrations inside each package were measured with a $CO_2/O_2$ analyser (CheckPoint, PBI Dansensor, Italy). Changes in gas composition values were measured over the entire period of the trial and are expressed as v/v kPa. To avoid modification of headspace gas composition due to gas sampling, the analyser introduced the same quantity of air that it removed for the analysis. To prevent gas leakage during measurements, an adhesive septum (Septum white 15 mm, Dansensor, Italy) was placed on the film surface. Calibration was performed using air (Aday & Caner, 2011). The results are expressed based on the average of three replicates.

Table 1. Initial gas composition and film characteristics of the packages.

Tabla 1. Composición inicial de gas y características del papel film de los embalajes.

| Treatment | Modified atmosphere | Initial gas composition | Film packaging (25 µm) | $O_2$TR (ASTM F2622-08) at 23°C and 50% RH | $CO_2$TR (ASTM F2476-05) at 23°C and 50% RH |
|---|---|---|---|---|---|
| A | Passive | 20.8% $O_2$ + 0.2% $CO_2$ + 78% $N_2$ | Biodegradable and compostable | 3000 | 44113 |
| B | Active | 10% $O_2$ + 10% $CO_2$ + 80% $N_2$ | Biodegradable and compostable | 3000 | 44113 |
| C | Passive | 20.8% $O_2$ + 0.2% $CO_2$ + 78% $N_2$ | PP | 1456 | 4616 |
| D | Active | 10% $O_2$ + 10% $CO_2$ + 80% $N_2$ | PP | 1456 | 4616 |
| E | Control | 20.8% $O_2$ + 0.2% $CO_2$ + 78% $N_2$ | *Macro-perforated (PP with 6 mm holes) | 1456 | 4616 |

Note: *Due to the presence of macro holes (6 mm in diameter) in the film, no atmospheric modification was observed in the control; the film had only a mechanical protection function.

*Debido a la presencia de macroagujeros (6 mm de diámetro) en el papel film, no se observó ninguna modificación atmosférica en el control, el papel film únicamente tuvo una función de protección mecánica.

### *Quality measurements*

The weight (i.e. water) loss (% from original weight) from each tray was measured using an electronic balance (SE622, WVR Science Education, USA) with an accuracy of 0.01 g. The results are expressed as an average of three replicates.

Soluble solids content (SSC) was determined in the juice (from three trays randomly chosen for each treatment) using a digital refractometer (Atago PR-101, Atago, Japan) at 20°C. Two readings (30 fruits) were taken for each fruit and averaged, and results are expressed as °Brix.

Titratable acidity (TA) was determined by titration (Titritino 702, Metrohm, Switzerland) with 0.1 N NaOH up to pH 8.1, using 10 ml of diluted juice in distilled $H_2O$, and the results are expressed as meq/L.

Colour was measured on the first 15 sound, non-mouldy fruits from each tray (three trays were randomly chosen for each package). The mean of 30 measurements was used for data analysis. Colour was measured on the side of a slightly flattened whole fruit using a tristimulus colour analyser (Chroma Meter, Model CR-400, Minolta, Germany) equipped with a measuring head with measuring area of 8 mm, and is represented by lightness (L), chroma (C) and hue angle (h), in accordance with the CIE Lab system colorimeter (Colortec PCM, Clinton, NJ, USA).

### *Fruit volatile components*

Volatile components were analysed by the automatic headspace solid-phase dynamic extraction (HS-SPDE) technique combined with a gas chromatograph-mass spectrophotometer (GC-MS) and a CTC Analytics Combi Pal autosampler. The samples (three trays, 0.150 kg for each treatment) were blended into a fine puree, of which 3 g was placed in a 20 ml headspace glass vial (Brown Chromatography Supplies, Kreuzwertheim, Germany) with 5 g of sodium chloride to increase extraction recovery. After equilibration at 35°C for 15 min, the volatile components were extracted with SPDE fibre (SPNdl-01/AC-50-56, PDMS + 10% active charcoal, 50 μm, 57 mm) exposed to the headspace for 30 min. The fibre was then introduced to the injector of the GC for desorption at 250°C for 3 min in splitless mode. Analysis of volatile compounds was performed using an Agilent 6890 gas chromatograph equipped with a Restek Rxi-1 MS column (30 m × 0.25 mm ID × 0.25 μm film thickness) and a mass-spectrometer detector (Agilent 5973). The initial oven temperature was held at 0°C for 2 min, increased by 5°C/min to 100°C, again by 4°C/min to 230°C and held for 10 min. Mass spectra were scanned at m/z 35–300 and volatile compounds were identified by comparing data with the Wiley Library (Wiley 9th) and retention times. The system was set up through the injection of a standard mixture solution (terpenes/terpenoids, esters, aldehydes, hydrocarbons) in methanol at a concentration of 0.01 μg/μl for each of the following components: hexanal, isoamyl acetate, camphene, beta-pinene, limonene, gamma-terpinene, delta-3-carene, dodecane, menthol, carvone, alpha-copaene, pentadecane, hexadecane and heptadecane (Sigma–Aldrich Co., Milan, Italy). Concentrations of the compounds were increased and system linearity was verified under operating conditions, and good correlation coefficients ($r \geq 0.85$) were found. The repeatability of the method (Peano et al., 2013) was investigated by performing three different injections from each 0.150 kg tray. Quantitative analysis was performed using the external standard calibration, with a focus on checking the retention time of each single component identified by mass spectra.

### *Statistical analysis*

The data were subjected to one-way analysis of variance (ANOVA) with Tukey's test to determine the significance of differences of means between the groups, using the Software IBM-SPSS.20 (2013). The differences were considered significant at $P < 0.05$.

## Results and discussion

### *Headspace gas composition*

The initial atmospheric composition in the packages changed rapidly for all treatments, and both films were able to manage MAP conditions. The exchange area through the film packages (550 cm$^2$) was kept constant, so the evolution of the internal atmosphere inside them was influenced by fruit respiration and the permeability of films to $O_2$ and $CO_2$ (Beaudry, Cameron, Shirazi & Dostallange, 1992). The lowest $O_2$ and highest $CO_2$ partial pressures were achieved gradually with the biodegradable and compostable film (treatments A and B), which also improved the storability of raspberries at the highest temperature (96 h). Raspberries wrapped in the biodegradable and compostable film in active MAP (treatment B) maintained higher $CO_2$ levels at all time points than the produce undergoing corresponding passive MAP (treatment A). The increase in storage temperature after 48 h caused an increase in fruit respiration rate; this occurred at a faster rate than the increase in permeation through the biodegradable film, resulting in depletion of $O_2$ and an increase in $CO_2$ in both passive and active MAP packages (treatments A and B). In no case were any injurious gas levels observed. At the end of the storage period, 3.6 and 4.9 kPa $O_2$ and 24.4 and 25.9 kPa $CO_2$ was observed for treatments A and B, respectively. Steady-state $O_2$ and $CO_2$ levels and the time to reach these levels were a function of film characteristics, produce respiration rate and initial atmospheric composition (Kader, Zagory, Kerbel & Wang, 1989). In our study, this condition was achieved with treatment A only after 72 h of storage (9.4 kPa $O_2$ and $CO_2$), but it was immediately lost. Due to the presence of macro holes (6 mm diameter) in the film, no atmospheric modification was observed in the control (treatment E).

### *Quality measurements*

Weight loss (i.e. water) in raspberries is generated by the high metabolic activity (i.e. respiration and transpiration rates) of the fruits. However, in our study, weight loss was not a limiting factor for berry quality, even when fruits were subjected to metabolic stress due to a change in temperature (96 h), because weight loss below 6–8% is considered the limit of marketability (Haffner et al., 2002; Nunes, Emond & Brecht, 2003). Weight loss (Figure 1) with the perforated film (treatment E) ranged from 1 to 2% and in the MAP packages from 0.1 to 0.2% (data not shown). All MAP treatments maintained a good state of hydration of the fruits, and no fungal decay was detected in the packages. Sweetness and acidity components affect the flavour and the taste of raspberry fruits, but this ratio (SSC/TA) remained almost unchanged in all treatments (0.6–0.9) during the storage period (data not shown). The SSC of raspberries at harvest was 11.0° Brix (Table 2); after 48 h of storage at low temperature (1 ± 1°C) it was subsequently affected by storage treatment. All treatments showed a significant reduction in SSC

Table 2. Changes in soluble solid content, titratable acidity and colour of cv. Himbo Top in storage at 1 ± 1°C followed by 18 ± 1°C.

Tabla 2. Cambios en el contenido de sólidos solubles, acidez valorable y color de cv. Himbo Top en el almacén a 1 ± 1°C seguido de 18 ± 1°C.

| Hours | Treatment | SSC (°Brix) | TTA (meq/l) | L | C | h |
|---|---|---|---|---|---|---|
| 48 | Harvest | 11.0 ± 0.1$^{a}$ | 15.6 ± 0.3$^{a}$ | 28.4 ± 2.7$^{a}$ | 31.8 ± 5.8$^{a}$ | 0.40 ± 0.1$^{a}$ |
| | A | 10.2 ± 0.1$^{b}$ | 14.6 ± 0.1$^{a,b}$ | 26.9 ± 2.6$^{a,b}$ | 26.1 ± 3.9$^{b,c}$ | 0.35 ± 0.1$^{b,c,d}$ |
| | B | 10.0 ± 0.1$^{b}$ | 14.6 ± 0.1$^{a,b}$ | 27.6 ± 2.4$^{a}$ | 26.3 ± 2.9$^{b,c}$ | 0.37 ± 0.1$^{a,b,c}$ |
| | C | 9.4 ± 0.2$^{c}$ | 13.1 ± 0.9$^{c}$ | 25.6 ± 1.2$^{b}$ | 27.7 ± 1.1$^{b}$ | 0.39 ± 0.1$^{a,b}$ |
| | D | 9.5 ± 0.1$^{c}$ | 13.5 ± 0.2$^{b,c}$ | 24.7 ± 2.6$^{b}$ | 26.0 ± 3.0$^{b,c}$ | 0.31 ± 0.1$^{d}$ |
| | E | 8.6 ± 0.0$^{d}$ | 12.9 ± 0.7$^{c}$ | 25.2 ± 1.3$^{b}$ | 23.3 ± 2.5$^{c}$ | 0.33 ± 0.0$^{c,d}$ |
| 96 | Harvest | 11.0 ± 0.2$^{a}$ | 15.6 ± 0.3$^{a}$ | 28.4 ± 2.7$^{a}$ | 31.8 ± 5.8$^{a}$ | 0.40 ± 0.1$^{a}$ |
| | A | 9.7 ± 0.1$^{b}$ | 14.6 ± 0.9$^{a,b}$ | 25.9 ± 1.6$^{b}$ | 24.7 ± 2.8$^{b}$ | 0.29 ± 0.2$^{b}$ |
| | B | 9.6 ± 0.1$^{b}$ | 13.8 ± 0.2$^{b}$ | 25.8 ± 1.5$^{b}$ | 25.8 ± 3.0$^{b}$ | 0.28 ± 0.3$^{b}$ |
| | C | – | – | – | – | – |
| | D | – | – | – | – | – |
| | E | – | – | – | – | – |

Note: Means in a column followed by different superscript letters are significantly different at $P \leq 0.05$ according to Tukey's test.

Los promedios en la columna seguidos de diferentes letras son significativamente distintos p ≤ 0,05 según el test de Tukey.

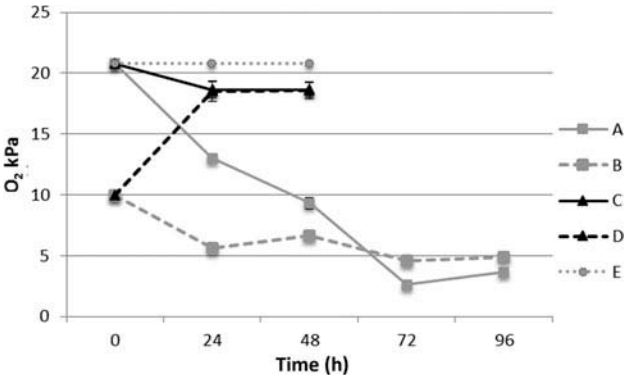

Figure 1. Levels of $O_2$ in the headspace of raspberry cv. Himbo Top stored in MAP.

Figura 1. Niveles de $O_2$ en la parte superior de la frambuesa cv. Himbo Top almacenada en MAPs.

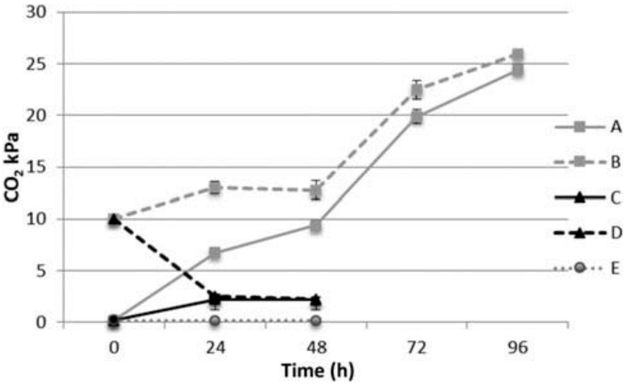

Figure 2. Levels of $CO_2$ in the headspace of raspberry cv. Himbo Top stored in MAP.

Figura 2. Niveles de $CO_2$ en la parte superior de la frambuesa cv. Himbo Top almacenada en MAPs.

values when compared with harvest time, probably due to respiration activity, although respiration activity was not monitored. The highest reduction was observed in control fruits (E treatment) while among MAP conditions, reduction was higher in fruits wrapped in PP film, under both passive and active atmospheres (treatments C and D). After 96 h of storage with change in temperature, SSC content increased but no statistically significant differences were observed between treatments A and B. The initial TA value was 15.6 meq/l and during storage this decreased under all treatments, in agreement with Haffner et al. (2002). In regard to sugars, reduction in TA was probably caused by fruit respiration. After 48 h at 1 ± 1°C, the biodegradable and compostable film (treatments A and B) showed the highest TA value (14.6 meq/l) because these treatments had the highest $CO_2$ headspace concentration, corroborating the results of Malhotra and Prasad (1999). Colour changes (Table 2) were significantly affected by the treatments, with decreasing lightness (L) and increasing redness as indicated by decreasing chroma (C) and hue angle (h) values, respectively. After 48 h, fruits became darker and, according to the literature (Almenar et al., 2007; Caner, Aday & Demir, 2008), all treatments show L values lower than those at harvest (28.4). A decrease in L value reflects darkening of fruit, probably due to the formation of dark compounds by the oxidation of phenolics, and indicates that the senescence process is occurring. Treatments A and B showed the highest L values as a consequence of the high $CO_2$ concentration observed in the headspace of the biodegradable film (Figure 2), confirming the findings of Haffner et al. (2002). After 96 h, similar L values (25.9 and 25.8, respectively) were found. During storage, fruits became less vivid than at harvest (lower chroma, 31.80), and this trend was more evident for fruit packaged with the perforated film (E treatment) which showed the lowest C value (23.3) after 48 h. This can be explained by oxidative browning reaction, as observed in other species in the presence of $O_2$ (Aday & Caner, 2011; Nunes et al., 2009). The h value is directly related to humidity during storage (GonCalves et al., 2007) decreasing at room temperature with low RH values; at the end of storage (96 h), no statistically significant differences were observed between treatments A and B.

## *Fruit volatile components*

There are approximately 200 aromatic volatile components of raspberries reported in the literature (Klesk, Qian &

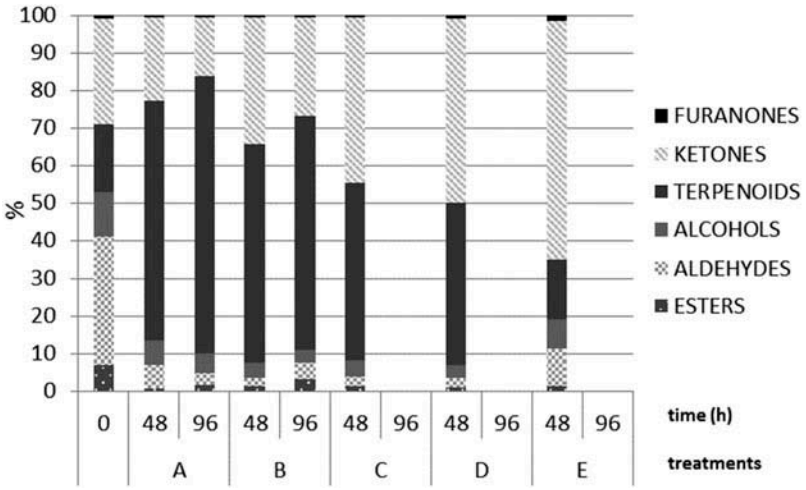

Figure 3. Major aroma compounds evaluated in raspberries cv. Himbo Top stored in MAPs.

Figura 3. Principales compuestos aromáticos evaluados en frambuesa cv. Himbo Top almacenada en MAPs.

Martin, 2004); in this study on cv. Himbo Top, 54 volatile compounds were identified from six different classes. In fresh fruits, as shown in Figure 3, aldehydes and ketones were the most represented (34 and 28%, respectively), followed by terpenes (18%), alcohols (12%), esters (7%) and furans (1%). The selected aroma substances for each aromatic class were quantified daily, but the most interesting findings were observed after 48 and 96 h. Mean values (µg/kg) from GC analyses of triplicate extractions and standard deviations are reported for all aroma compounds in Tables 3–5.

Volatile esters (Table 3) are generated by the esterification of alcohols and acyl-CoAs derived from both fatty acid and amino acid metabolism, in a reaction catalysed by the enzyme alcohol-o-acyltransferase. The effect of $CO_2$ on ester production is unclear (Beaudry, 1999). In our study, butanoate and acetate esters were the two main groups of compound found in cv. Himbo Top. The total level of esters at harvest was 3.84 µg/kg. After 48 h of storage at 1 ± 1°C, all treatments showed a decrease in the total amount of esters, while at 96 h with a change in temperature to 18 ± 1°C, and in agreement with the literature (Ulrich, Komes, Olbricht & Hoberg, 2007), the levels increased probably due to water loss (data not shown) and increase in $CO_2$ concentration inside the packages wrapped with the biodegradable film both in passive and active MAP (treatments A and B) (Ke, Zhou & Kader, 1994). Some compounds, such as 2-hexenyl acetate, phenethyl acetate and ethyl benzoate, were not found in the fruits at harvest or after 48 h of storage at 1 ± 1°C; they were instead synthesized at 18 ± 1°C. At the end of the storage period, the highest concentration of esters (6.40 µg/kg) was observed with treatment B; isoamyl acetate (3.20 µg/kg) was the prevalent chemical component, being responsible for a fruity odour described as being similar to that of banana or pear (Surburg & Panten, 2005).

As observed in previous research (Dixon & Hewett, 2001; Ozcan & Barringer, 2011; Peano et al., 2013), the levels of aldehydes (Table 3) changed during the storage period. According to Jetti, Yang, Kurnianta, Finn and Qian (2007), (E)-2-hexenal and hexanal are the major components in raspberries at harvest (16.57 and 4.9 µg/kg, respectively). After 48 h at low temperature (1 ± 1°C), all treatments showed lower values compared with those at harvest (22.90 µg/kg), ranging from a maximum of 7.97 µg/kg (treatment A) to a minimum of 1.86 µg/kg (treatment C), suggesting the role of the surrounding atmosphere. A total of four alcohols were identified and quantified in cv. Himbo Top (Table 4); their chemical composition was dominated by 3-hexanol and 2-heptanol (2.01 and 6.02 µg/kg, respectively at harvest). After 48 h of storage at 1 ± 1°C, all treatments showed a decrease in total content from that measured at harvest (8.33 µg/kg). The availability of alcohol compounds is thought to be one of the limiting factors for ester production (Beekwilder et al., 2004) and, as suggested by Ke et al. (1994), low $O_2$ and high $CO_2$ concentrations are responsible for the accumulation of alcohols, which leads to the production of ethyl esters and the reduction in other esters. In our study, the highest total concentration of alcohols (12.49 µg/kg) was observed in fruits wrapped in the biodegradable film under passive MAP (A treatment), due to the combined effect of change in temperature to 18 ± 1°C (96 h) and high $CO_2$ concentration in the packages, which, moreover, corresponded to the lowest total ester level found.

The total content of terpene compounds in the fresh fruits was 12.28 µg/kg. These are mainly represented by monoterpenes (C10) and sesquiterpenes (C15) that ranged between a minimum of 16% (8.07 µg/kg) with the E treatment to 74% (174.12 µg/kg) with the A treatment. These are responsible for the aromatic notes of flowers and herbs (Morales et al., 2014), and all MAP treatments showed an increase in these compared with harvest values.

After 48 h, under both passive and active MAP conditions (A and B, respectively), the raspberries that were wrapped in the non-commercial and biodegradable film already showed twice the level of terpenes (77.30 and 75.07 µg/kg, respectively) compared with those in the corresponding samples wrapped in PP film (35.16 and 38.80 µg/kg).

Volatility increased with temperature change and, at the end of storage, the highest levels were found for the A treatment (174.12 µg/kg). As described for other species, the high content of ketones and terpenes observed in stored fruits combined with a lower SSC/TA ratio could yield more acidic, green and piney flavours compared with fresh fruits (Beaulieu & Lea, 2003). The fruit volatile composition of cv. Himbo Top raspberries was

Table 3. Esters and aldehydes in cv. Himbo Top raspberries under MAP storage.

Tabla 3. Ésteres y aldehídos en frambuesas cv. Himbo Top bajo almacenamiento MAP.

| | | Treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | A | | B | | C | | D | | E | |
| | | Hours | | | | | | | | | | |
| Aroma component (μg/kg) | | 0 | 48 | 96 | 48 | 96 | 48 | 96 | 48 | 96 | 48 | 96 |
| Esters | Ethyl butanoate | 0.72 ± 0.01 | 0.0 ± 0.0 | 0.27 ± 0.01 | 0.0 ± 0.0 | 0.46 ± 0.01 | 0.16 ± 0.01 | – | 0.16 ± 0.01 | – | 0.18 ± 0.0 | – |
| | Butyl acetate | 0.98 ± 0.01 | 0.15 ± 0.01 | 0.0 ± 0.0 | 0.52 ± 0.01 | 0.76 ± 0.01 | 0.20 ± 0.01 | – | 0.17 ± 0.0 | – | 0.17 ± 0.0 | – |
| | Isoamyl acetate | 0.24 ± 0.01 | 0.12 ± 0.01 | 1.95 ± 0.2 | 0.32 ± 0.01 | 3.20 ± 0.1 | 0.05 ± 0.0 | – | 0.08 ± 0.0 | – | 0.02 ± 0.0 | – |
| | Ethyl caproate | 0.14 ± 0.00 | 0.12 ± 0.01 | 0.60 ± 0.03 | 0.19 ± 0.01 | 0.37 ± 0.01 | 0.11 ± 0.0 | – | 0.13 ± 0.0 | – | 0.16 ± 0.01 | – |
| | 3-Hexenyl acetate | 1.77 ± 0.2 | 0.30 ± 0.01 | 0.79 ± 0.02 | 0.69 ± 0.01 | 0.78 ± 0.01 | 0.44 ± 0.01 | – | 0.34 ± 0.0 | – | 0.21 ± 0.01 | – |
| | 2-Hexenyl acetate | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.04 ± 0.0 | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – |
| | Phenethyl acetate | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.26 ± 0.01 | 0.0 ± 0.0 | 0.07 ± 0.0 | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – |
| | Ethyl benzoate | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.04 ± 0.0 | 0.0 ± 0.0 | 0.71 ± 0.01 | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – |
| | Total | **3.84** | **0.69** | **3.90** | **1.73** | **6.40** | **0.96** | – | **0.88** | – | **0.75** | – |
| Aldehydes | (E)-2-Hexenal | 16.57 ± 1.2 | 6.35 ± 0.9 | 2.82 ± 0.2 | 1.98 ± 0.1 | 6.71 ± 0.2 | 0.88 ± 0.02 | – | 1.28 ± 0.1 | – | 1.11 ± 0.2 | – |
| | Hexanal | 4.9 ± 0.2 | 1.4 ± 0.1 | 3.7 ± 0.3 | 0.4 ± 0.01 | 1.0 ± 0.1 | 0.3 ± 0.01 | – | 0.4 ± 0.01 | – | 0.5 ± 0.05 | – |
| | Heptanal | 0.1 ± 0.0 | 0.1 ± 0.01 | 0.0 ± 0.0 | 0.1 ± 0.01 | 0.1 ± 0.1 | 0.1 ± 0.01 | – | 0.1 ± 0.01 | – | 2.8 ± 0. 1 | – |
| | 2,4-Hexadienal | 0.28 ± 0.01 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – | 0.10 ± 0.01 | – |
| | Benzaldehyde | 0.1 ± 0.0 | 0.1 ± 0.0 | 0.1 ± 0.01 | 0.1 ± 0.01 | 0.1 ± 0.01 | 0.1 ± 0.01 | – | 0.1 ± 0.01 | – | 0.1 ± 0.01 | – |
| | Nonanal | 0.7 ± 0.01 | 0.0 ± 0.0 | 0.2 ± 0.01 | 0.2 ± 0.01 | 0.2 ± 0.01 | 0.3 ± 0.01 | – | 0.2 ± 0.01 | – | 0.3 ± 0.01 | – |
| | Decanal | 0.3 ± 0.01 | 0.0 ± 0.0 | 0.3 ± 0.01 | 0.2 ± 0.01 | 0.2 ± 0.01 | 0.2 ± 0.01 | – | 0.2 ± 0.01 | – | 0.2 ± 0.01 | – |
| | Total | **22.90** | **7.97** | **7.05** | **3.01** | **8.28** | **1.86** | – | **2.24** | – | **5.10** | – |

Notes: Biodegradable and compostable film (A), biodegradable and compostable film + gas (B), polypropylene (PP) film (C), PP film + gas (D), PP perforated film (E).

Papel film biodegradable y compostable (A), Papel film + gas biodegradable y compostable (B), Papel film de polipropileno (C), Papel film + gas de polipropileno (D), Papel film de polipropileno perforado (E).

Table 4. Alcohols and terpenes in cv. Himbo Top raspberries under MAP storage.

Tabla 4. Alcoholes y terpenos en frambuesas cv. Himbo Top bajo almacenamiento MAP.

| | | Treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | A | | B | | C | | D | | E | |
| | | Hours | | | | | | | | | | |
| Aroma component (µg/kg) | | 0 | 48 | 96 | 48 | 96 | 48 | 96 | 48 | 96 | 48 | 96 |
| Alcohols | 3-Hexanol | 2.01 ± 0.3 | 1.88 ± 0.2 | 5.07 ± 0.2 | 0.55 ± 0.03 | 2.66 ± 0.2 | 0.27 ± 0.02 | – | 0.16 ± 0.0 | – | 0.96 ± 0.02 | – |
| | 2-Heptanol | 6.02 ± 0.4 | 5.84 ± 0.2 | 6.91 ± 0.3 | 4.43 ± 0.3 | 3.77 ± 0.3 | 2.99 ± 0.1 | – | 2.77 ± 0.1 | – | 2.89 ± 0.1 | – |
| | Butanol | 0.25 ± 0.01 | 0.06 ± 0.0 | 0.34 ± 0.02 | 0.10 ± 0.01 | 0.14 ± 0.01 | 0.11 ± 0.01 | – | 0.08 ± 0.01 | – | 0.11 ± 0.001 | – |
| | Fenchyl alcohol | 0.05 ± 0.0 | 0.00 ± 0.0 | 0.18 ± 0.00 | 0.06 ± 0.01 | 0.13 ± 0.01 | 0.04 ± 0.0 | – | 0.05 ± 0.0 | – | 0.00 ± 0.0 | – |
| | Total | **8.33** | **7.78** | **12.49** | **5.13** | **6.69** | **3.42** | – | **3.05** | – | **3.97** | – |
| Terpenes | β-Pinene | 0.0 ± 0.0 | 0.1 ± 0.01 | 2.1 ± 0.1 | 1.05 ± 0.01 | 1.5 ± 0.01 | 0.52 ± 0.01 | – | 0.56 ± 0.01 | – | 0.11 ± 0.01 | – |
| | Myrcene | 0.20 ± 0.01 | 0.3 ± 0.01 | 6.5 ± 0.3 | 1.72 ± 0.01 | 3.5 ± 0.1 | 0.59 ± 0.01 | – | 0.62 ± 0.01 | – | 0.26 ± 0.01 | – |
| | α-Phellandrene | 0.21 ± 0.01 | 40.3 ± 0.01 | 51.0 ± 1.5 | 21.02 ± 1.2 | 33.3 ± 1.2 | 7.71 ± 0.2 | – | 9.10 ± 0.8 | – | 1.00 ± 0.1 | – |
| | α-Terpinene | 0.31 ± 0.01 | 0.3 ± 0.02 | 2.2 ± 0.1 | 1.17 ± 0.01 | 1.8 ± 0.01 | 0.65 ± 0.01 | – | 0.70 ± 0.03 | – | 0.22 ± 0.01 | – |
| | p-Cymene | 0.16 ± 0.01 | 0.3 ± 0.0 | 9.7 ± 0.9 | 3.82 ± 0.3 | 7.4 ± 0.4 | 1.94 ± 0.01 | – | 2.06 ± 0.1 | – | 0.48 ± 0.01 | – |
| | Limonene | 0.39 ± 0.02 | 0.8 ± 0.01 | 5.2 ± 0.2 | 2.29 ± 0.1 | 3.2 ± 0.1 | 0.98 ± 0.01 | – | 0.91 ± 0.02 | – | 0.60 ± 0.01 | – |
| | Sabinene | 0.13 ± 0.01 | 10.3 ± 0.01 | 22.7 ± 1.1 | 9.78 ± 0.6 | 15.3 ± 0.9 | 3.70 ± 0.1 | – | 4.08 ± 0.2 | – | 0.45 ± 0.01 | – |
| | Linalool | 0.00 ± | 0.3 ± 0.01 | 1.4 ± 0.1 | 0.37 ± 0.01 | 0.6 ± 0.01 | 0.0 ± 0.0 | – | 0.18 ± 0.01 | – | 0.26 ± 0.01 | – |
| | Longicyclene | 0.05 ± 0.00 | 0.6 ± 0.01 | 5.3 ± 0.2 | 1.45 ± 0.1 | 2.3 ± 0.1 | 0.10 ± 0.01 | – | 0.65 ± 0.01 | – | 0.07 ± 0.01 | – |
| | Caryophyllene | 1.29 ± 0.1 | 1.7 ± 0.2 | 12.1 ± 1.0 | 3.94 ± 0.1 | 6.9 ± 0.2 | 2.81 ± 0.01 | – | 2.71 ± 0.1 | – | 0.49 ± 0.02 | – |
| | δ-Cadinene | 7.44 ± 0.9 | 0.1 ± 0.01 | 0.2 ± 0.01 | 0.08 ± 0.0 | 0.1 ± 0.0 | 0.08 ± 0.01 | – | 0.06 ± 0.0 | – | 0.07 ± 0.01 | – |
| | Others | 2.08 ± 0.1 | 22.39 ± 0.02 | 55.84 ± 2.1 | 28.37 ± 1.9 | 43.43 ± 2.1 | 16.09 ± 0.4 | – | 17.17 ± 0.9 | – | 4.07 ± 0.2 | – |
| | Total | **12.28** | **77.30** | **174.12** | **75.07** | **118.98** | **35.16** | – | **38.80** | – | **8.07** | – |

Notes: Biodegradable and compostable film (A), biodegradable and compostable film + gas (B), polypropylene (PP) film (C), PP film + gas (D), PP perforated film (E).

Papel film biodegradable y compostable (A), Papel + gas film biodegradable y compostable (B), Papel film de polipropileno (C), Papel film + gas de polipropileno (D), Papel film de polipropileno perforado (E).

Table 5. Ketones and furanones in cv. Himbo Top raspberries under MAP storage.

Tabla 5. Cetonas y furanonas en frambuesas cv. Himbo Top bajo almacenamiento MAP.

| | | Treatment | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | A | | B | | C | | D | | E | |
| | | Hours | | | | | | | | | | |
| Aroma component (μg/kg) | | 0 | 48 | 96 | 48 | 96 | 48 | 96 | 48 | 96 | 48 | 96 |
| Ketones | 2-Heptanone | 0.79 ± 0.1 | 0.72 ± 0.01 | 0.00 ± 0.0 | 1.51 ± 0.2 | 1.99 ± 0.3 | 2.24 ± 0.5 | – | 1.87 ± 0.9 | – | 2.05 ± 0.2 | – |
| | P-methylcyclo-hexanone | 0.00 ± 0.0 | 0.11 ± 0.01 | 0.00 ± 0.0 | 0.05 ± 0.01 | 0.00 ± 0.0 | 0.00 ± 0.0 | – | 0.08 ± 0.01 | – | 0.08 ± 0.3 | – |
| | Isophorone | 0.00 ± 0.0 | 0.04 ± 0.01 | 0.00 ± 0.0 | 0.04 ± 0.01 | 0.00 ± 0.0 | 0.05 ± 0.01 | – | 0.07 ± 0.0 | – | 0.05 ± 0.2 | – |
| | Acetophenone | 0.10 ± 0.01 | 0.15 ± 0.01 | 0.23 ± 0.01 | 0.28 ± 0.01 | 0.23 ± 0.0 | 0.22 ± 0.01 | – | 0.31 ± 0.01 | – | 0.18 ± 0.01 | – |
| | Nonanone | 0.10 ± 0.01 | 0.28 ± 0.0 | 1.44 ± 0.2 | 0.37 ± 0.01 | 0.61 ± 0.01 | 0.19 ± 0.01 | – | 0.18 ± 0.01 | – | 0.26 ± 0.01 | – |
| | β-Ionone | 13.00 ± 0.9 | 20.04 ± 1.1 | 27.32 ± 1.1 | 27.83 ± 1.9 | 37.13 ± 1.3 | 19.85 ± 0.8 | – | 29.47 ± 0.02 | – | 22.82 ± 1.1 | – |
| | Damascenone | 0.12 ± 0.01 | 0.12 ± 0.0 | 0.09 ± 0.01 | 0.20 ± 0.0 | 0.15 ± 0.01 | 0.13 ± 0.01 | – | 0.13 ± 0.01 | – | 0.13 ± 0.01 | – |
| | α-Ionone | 3.83 ± 0.01 | 4.49 ± 1.1 | 7.06 ± 1.1 | 13.03 ± 1.2 | 8.64 ± 0.9 | 9.45 ± 0.9 | – | 9.89 ± 0.9 | – | 5.00 ± 0.2 | – |
| | Dihydro-β-ionone | 0.80 ± 0.01 | 1.35 ± 0.5 | 0.64 ± 0.02 | 0.50 ± 0.02 | 1.82 ± 0.1 | 0.79 ± 0.2 | – | 2.42 ± 0.2 | – | 1.80 ± 0.1 | – |
| | Total | **18.74** | **27.30** | **36.78** | **43.81** | **50.57** | **32.92** | – | **44.41** | – | **32.37** | – |
| Furanones | 2-Amylfuran | 0.10 ± 0.01 | 0.0 ± 0.0 | 0.09 ± 0.01 | 0.0 ± 0.0 | 0.12 ± 0.00 | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – |
| | 5-Ethyldihydro-2(3H)-furanone | 0.20 ± 0.01 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – | 0.0 ± 0.0 | – |
| | Theaspirane | 0.51 ± 0.02 | 0.57 ± 0.01 | 0.93 ± 0.00 | 0.71 ± 0.02 | 1.01 ± 0.2 | 0.57 ± 0.03 | – | 0.59 ± 0.02 | – | 0.75 ± 0.02 | – |
| | Total | **0.81** | **0.57** | **1.02** | **0.71** | **1.13** | **0.57** | – | **0.59** | – | **0.75** | – |

Notes: Biodegradable and compostable film (A), biodegradable and compostable film + gas (B), polypropylene (PP) film (C), PP film + gas (D), PP perforated film (E).

Papel film biodegradable y compostable (A), Papel film + gas biodegradable y compostable (B), Papel film de polipropileno (C), Papel film + gas de polipropileno (D), Papel film de polipropileno perforado (E).

dominated by ketones that ranged between 28.0 and 63.0% of the total aroma classes throughout the storage period (Figure 3). Raspberry cv. Himbo Top is strongly characterized by the presence of α- and β-ionones (3.83 and 13.00 μg/kg, respectively at harvest) confirming previous studies on other cultivars (Morales et al., 2014). The characteristic raspberry ketone 4-(4-hydroxyphenyl) butan-2-one reported in other studies on raspberry aroma (Robertson, Griffiths, Woodford & Birch, 1995) has not been reported to be present in cv. Himbo Top. For each treatment at both storage temperatures, β-ionone was the main compound responsible for increase in total components, ranging from a minimum value of 19.85 μg/kg in the C treatment to a maximum of 37.13 μg/kg in the B treatment (respectively after 48 and 96 h).

Furanones represent less than 1% of the total aroma components of stored raspberries and are known to be generated by carbohydrate metabolism. Despite the importance of furanones in fruit aroma, little is known about their biosynthesis and metabolism, except their instability. Furanones, in particular theaspirane, were found to be the most representative of all components during storage. Their content in fresh fruits was 0.57 μg/kg and seemed to be little affected by the different treatments during storage. Generally, atmospheric composition inside the packaging did not appear to affect total furanone content in raspberry cv. Himbo Top under different MAP conditions. As observed in previous studies on other fruits (Giuggioli, Girgenti, Baudino & Peano, 2014), the presence of furanones after 96 h, even when in limited concentrations (1.02 and 1.13 μg/kg, respectively, for treatments A and B), may indicate that the enzymatic system responsible for the synthesis of these compounds is still active at a temperature of 18 ± 1°C.

## Conclusions

Interaction between raspberry fruits, packaging film and the external environment (i.e. MAP conditions and storage temperature) has the potential to significantly influence the overall quality of the product. Considering all the qualitative parameters and aroma compounds measured, non-commercial biodegradable and compostable film showed the best results for storing fruits over a period of 96 h (48 h at 1°C followed by 48 h at 18°C). Among all aromatic components, terpenes and ketones underwent the greatest change during storage. The non-commercial biodegradable film used in this work can be proposed as a suitable packaging for raspberry fruits under both passive and active MAP conditions, due the more attractive colour of the berries and the similarity of aroma profiles, mainly dominated by terpenes. Packaging using film made from a renewable source such as that used in this work represents a promising solution to improving the marketability of these fruits.

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