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Novel combination of ethylene oxidisers to delay losses on postharvest quality, volatile compounds and sensorial analysis of tomato fruit

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ABSTRACT

Ethylene is a phytohormone naturally produced by plants and fruits (especially climacteric ones) all along the growing stage. During post-harvest, it is one of the main agents involved in the ripening of tomato fruits, even leading to severe quality losses. The aim of the present study was to determine, in tomato fruit, the possible effect of two different ethylene removal through oxidation mechanisms (ultraviolet light and $KMnO_4$ filters) on postharvest quality, antioxidant capacity, volatile compounds and sensory analysis carried out by experts. Two temperatures were selected for the use of this system, 8 °C and 20 °C. The use of this novel combination of techniques promoted the preservation of the physical and bioactive parameters analysed. A higher presence of volatile compounds related to early stages of fruit ripening was also observed in the treatments where ethylene removal was used, especially in the one where the complete system was used. It is also noteworthy that in the sensory analysis with a panel of experts, the fruit treated with the complete ethylene elimination system received a more favourable evaluation than those that did not incorporate it.

1. Introduction

Tomato (*Solanum lycopersicum* L.) is one of the most important agricultural crops, with 186,821,216 tons of worldwide production in 2020 (FAO, 2020). The organoleptic and nutritional quality of tomatoes, as well as their shelf life, are affected by several factors related to ripening and postharvest conditions such as temperature, conservation atmosphere, and microbial diseases during post-harvest storage. Fruit ripening is a complex, genetically-programmed process that ends with strong changes in colour, texture, flavour, aroma, and bioactive capacities of the fruit (Alexander & Grierson, 2002). With respect to the control of the conservation atmosphere in post-harvest preservation of fruit and vegetables, ethylene removal is one of the most critical aspects. Ethylene is a phytohormone that is naturally produced by climacteric fruit, and which promotes ripening processes. These ripening processes, although positive at the beginning of fruit growth, eventually lead to quality deterioration and product losses (Kader, 2011). Removing ethylene from the postharvest conservation environment could preserve important quality parameters such as pH, acidity, ascorbic acid concentration or antioxidant capacity during post-harvest conservation (Alonso-Salinas, Acosta-Motos, Núñez-Delicado, Gabaldón, & López-Miranda, 2022; Álvarez-Hernández, Martínez-Hernández, Castillejo, Martínez, & Artés-Hernández, 2021).

Ethylene removal is based on an oxidation-reduction process using an oxidising agent that dissociates ethylene into CO_2 and H_2O or blocking any pathway of ethylene signalling or production. According to Wei, Seidi, Zhang, Jin, and Xiao (2021), among the existing ethylene removal methods, the non-intrusive ones (which are those that do not come into contact with the products) are the most effective. In addition, the most important agents needed for a correct ethylene removal are classified by Wei et al. (2021) and Mansourbahmani, Ghareyazie, Zarinnia, Kalatejari, and Mohammadi (2018) as follows: palladium >

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$KMnO_4$ > 1-MCP > SA = $CaCl_2$ > UV-C. It should be noted that although the use of palladium is the most effective method in terms of ethylene oxidation, it is also the most expensive and therefore the most difficult to implement in the industrial sector.

Oxidation by potassium permanganate ($KMnO_4$) is the most recommended method for removing ethylene in terms of cost-effectiveness. This molecule changes in its colour from violet to dark brown at the saturation point, indicating the elimination of ethylene during the reaction (Janjarasskul & Suppakul, 2018; Pathak, 2019). $KMnO_4$ is introduced into porous materials with high adsorption power such as zeolite, vermiculite, alumina, sepiolite, or activated carbon to support this molecule (Alonso-Salinas et al., 2022; Álvarez-Hernández et al., 2018). These materials are extensively used in sachets placed in boxes during fruit transport. According to Salamanca, Balaguera-López, and Herrera (2014), the zeolite and $KMnO_4$ mixture was effective in the postharvest preservation of 'Chonto' tomato fruit, which showed the best postharvest performance with the lowest weight loss, a lagged increase in soluble solids content (SSC), and a higher firmness compared to control, indicating that the ripening process was delayed.

Ethylene oxidation by ultraviolet light stands out among existing methods because of its versatility, low cost and environmental friendliness. Pristijono et al. (2018), pointed out that UV-C treatment has been reported to have beneficial effect on maintaining postharvest quality of many horticultural products. Also, Liu, Cai, Lu, Han, and Ying (2012) and Mansourbahmani et al. (2018), showed that tomatoes treated with UV-C radiation maintained ascorbic acid, flavonoid and phenolic compound contents longer and in higher concentrations. No study has been found in the existing literature that analyses the effect of the combination of these two ethylene removal methods in depth.

The aim of this research was to study the combined effect of two different methods of ethylene removal, such as oxidation by $KMnO_4$ and photocatalysis by UV-C light, on the quality, shelf-life, organoleptic qualities and volatile compounds of tomatoes stored at two different temperatures (8 °C and 20 °C) over a 25 days storage period.

2. Materials and methods

2.1. Plant material

Forty kilograms of Rychka variety tomatoes (*Solanum lycopersicum* L.) were supplied by EXPOÁGUILAS, S·COOP. (Águilas, Murcia, Spain). Tomatoes were harvested in the traditional way and preserved at 8 °C for a few hours until laboratory transport for subsequent analysis. The final experimental tomatoes were selected paying attention to a homogeneous weight, size, colour, and ripening stage.

2.2. Experimental design

220 tomatoes were randomly distributed in five conservation chambers (CCs) of 150 L of volume (Eurofred Cool Head RCG200, Eurofred S.A., Barcelona, Spain).

According to Alonso-Salinas et al. (2022), the filters used were composed of $KMnO_4$ anchored to the active centre of sepiolite, which allowed for a better interaction of this oxidising substance with ethylene. The composition of the filters in terms of granulometry and other adsorbent substances was patented in Spain by the company "Nuevas Tecnologías Agroalimentarias" (KEEPCOOL) (Molina de Segura, Spain), patent No. 2548787 (2016). The adsorbing material was covered by a semi-permeable paper, which allowed the entry of air rich in ethylene and the output of air clean of this phytohormone. Conversely, this kind of paper prevented water or other particles to get into it and interfere with the process. Ethylene filters were installed inside an M-CAM 50 device (KEEPCCOL, Molina de Segura, Spain) which is an air-flow-forcing machine, to ensure an appropriate movement of the air through the filter. In addition, this device incorporates a photocatalytic ultraviolet light system UV-C (TUV 254 nm, Philips, Amsterdam, Netherland) to aid the $KMnO_4$ filters in the removal of ethylene. The ultraviolet light is focused on the air coming out of the filters, not on the fruit.

The experiment also studied the effect of the conservation temperatures. Two treatments at 8 °C and 20 °C were established, simulating near optimal and stressful temperatures, respectively.

In terms of ethylene removal, preservation temperature and relative humidity (90%), treatments were classified as follows:

- C: Cool temperature (8 °C). Control treatment.
- CF: Cool temperature (8 °C) + Filter.
- CFUv: Cool temperature (8 °C) + Filter + UV radiation.
- R: Room temperature (20 °C).
- RF: Room temperature (20 °C) + Filter.

The complete ethylene removal treatment was not applied to those CCs stored at 20 °C because it was observed that the device slightly raised the storage temperature. As it was not possible to maintain a stable temperature at 20 °C, it was decided not to apply it in order to respect the reproducibility of the study.

2.3. Physicochemical analysis

Soluble solid content (SSC), pH, and **titratable acidity (TA).** were measured on fruit samples using the method adapted from Zhang B., Peng, Zhang C., & Ma (2017) Twenty grams of tomato were taken and added to 20 mL of distilled water, then homogenized with a mixer (Ultra turrax T25, LabWare Wilmington, DE, USA) for 30 s. The homogenate was centrifuged at 3600×*g* for 10 min at 15 °C (Eppendorf Centrifuge 5810, Hamburg, Germany) and the supernatant was used to obtain SSC, pH, and TA.

The SSC was determined with a refractometer (Atago Manual master-α, Atago Tokyo, Japan) at 20 °C and expressed as a percentage. The pH was determined with a pH-meter (HI 2221, Hanna Instruments Eibar, Gipuzkoa, Spain).

The determination of TA was carried out by titration adapting the method described by Zhang, Peng, Zhang, Song, and Ma (2017) The concentration of acid in the sample was calculated and expressed as g L^{-1}.

Ripening index (RI) was determined by dividing SSC (expressed as %) by TA (expressed as %). The expression of this parameter is dimensionless.

Ascorbic acid analysis by 2,6-dichlorophenol indophenol method was adapted from the Nielsen (2017) method. The amount of ascorbic acid was determined and expressed as mg L^{-1} of tomato juice with the following formula ($F = 0.1$):

Equation 1:

$$Ascorbic\ acid\ \left(mg\ L^{-1}\right) = \frac{F\ x\ mL\ used\ of\ 2,6-DCF\ x\ 1000\ mL\ of\ juice}{Sample\ (mL)}$$

F = titer of dye (0.1 = mg ascorbic acid equivalent to 1.0 mL indophenol standard solution).

The total phenolic content (TPC) of fresh tomato extracts was determined coluorimetrically at 765 nm using the Folin-Ciocalteau reagent according to the method described by López-Miranda et al. (2016) TPC is expressed in mg of gallic acid equivalents per kilogram of fresh tomato (mg kg^{-1}).

The **ORAC** (antioxidant capacity) analyses were carried out on a Synergy HT multidetector microplate reader, from Bio-Tek Instruments, Inc. (USA), using 96-well polystyrene microplates with black sides and clear bottoms. Fluorescence was read through the clear bottom, with an excitation wavelength of 485 nm and an emission filter of 528 nm. The plate reader was controlled with KC4 software (3.4 version). The oxygen radical absorbance capacity was determined as described by López-Miranda et al. (2016). All reaction mixtures were prepared in

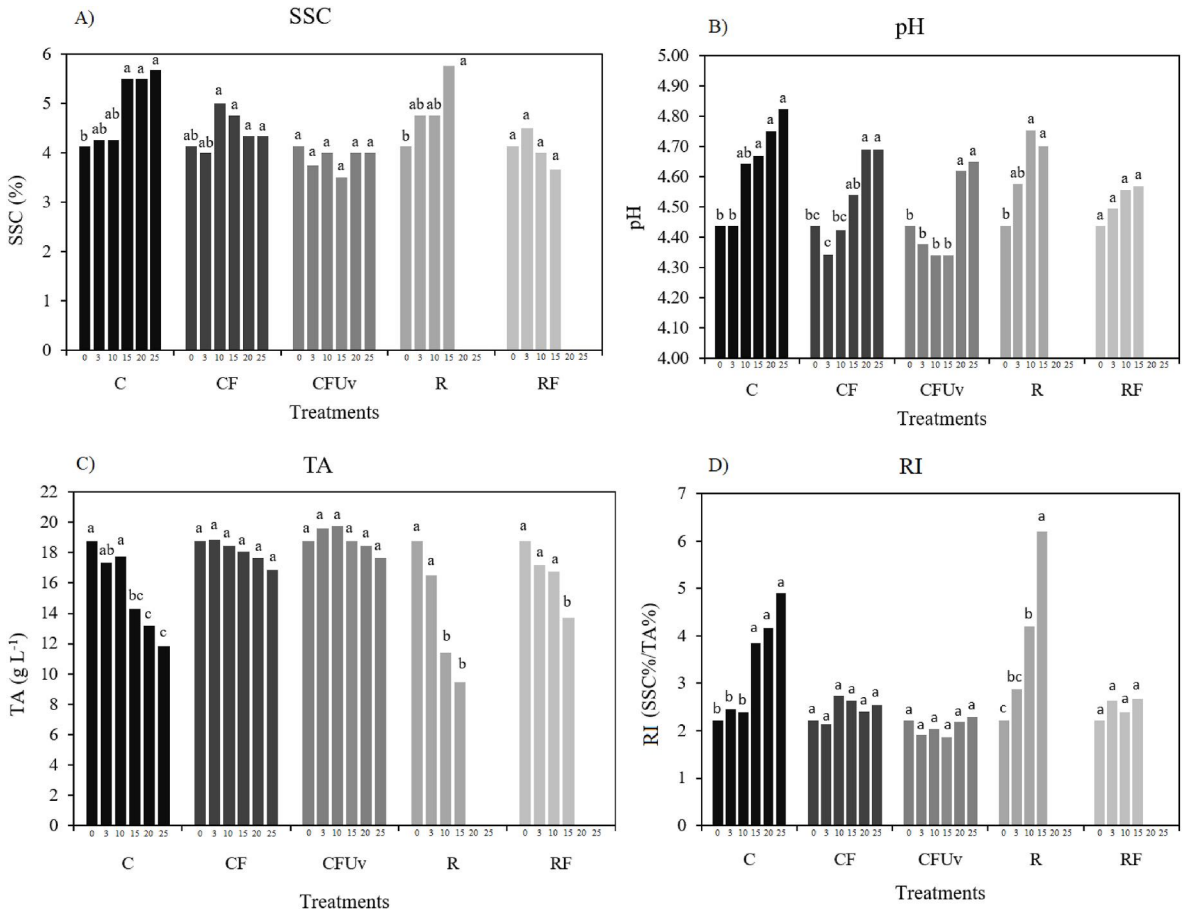

Fig. 1. Evolution during storage time of the ripening parameters in tomatoes subjected to different treatments (C, CF, CFUv, R and RF). The parameters measured were SSC expressed as percentage (a); pH (b); TA expressed as g L^{-1} (c) and RI expressed as the SSC (%)/TA (%) ratio (d). Different letters for each treatment represent statistically significant differences according to Tukey's test with the aim to see the evolution of each parameter on every treatment.

triplicate and at least three independent assays were performed for each sample. The results were expressed in μmol of Trolox equivalents per kilograms of fresh tomato (μmol kg^{-1}). The net area under the curve (AUC) corresponding to the sample was calculated by subtracting the AUC corresponding to the blank.

All the physicochemical analysis were carried out in triplicate for each tomato, on seven tomatoes per treatment and day throughout the entire storage period on the following days: 0, 3, 10, 15, 20 and 25.

2.4. Volatile compounds

The volatile composition of the tomato samples was determined using headspace solid phase micro-extraction (HS-SPME). After several preliminary tests to optimize the extraction system, 2 g of sample were weighted and added into a 15 mL vial with polypropylene caps and PTFE/silicone septa with 1 g of NaCl and 3 mL of ultrapure water. The vial was placed in an AOC-6000 Plus autosampler (Shimadzu Corporation, Kyoto, Japan) and, after 5 min of equilibration time, a 50/30 μm DVB/CAR/PDMS fibre (1 cm) was exposed to the sample headspace for 45 min at 40 °C (with agitation, 250 rpm). The separation and identification of compounds was performed with a GC2030 (Shimadzu Scientific Instruments, Inc., Columbia, MD, USA), in a Sapiens X5MS column (Teknokroma, Barcelona, Spain), 30 m × 0.25 mm i.d., 0.25 μm film thickness, and coupled with a mass spectrometer detector (TQ8040 NX triple quadrupole mass spectrometer; Shimadzu Scientific Instruments, Inc., Columbia, MD, USA). Only the single quadrupole acquisition mode was used on the TQ8040 NX (Q3 Scan; scan speed 5000 amu s^{-1}; mass range 40–400 m z^{-1}; event time 0.200 s). The oven temperature program was as follows: (i) initial temperature of 80 °C; (ii), increase of 3 °C min^{-1} up to 210 °C, and hold 2 min; (iii) increase of 20 °C min^{-1} up to 280 °C and, hold for 2 min the helium column head pressure was 38 kPa (constant linear velocity mode of 31 cm s^{-1}). The injector, ion source, and interface were set at 250, 230, and 280 °C, respectively. Helium was used as the carrier gas, and the column flow was 0.7 mL min^{-1}, with a 1:10 split ratio, and purge flow of 6 mL min^{-1}. Analyses of this parameter were carried out on days 0, 10, 15 and 20. According to Renard, Ginies, Gouble, Bureau, and Causse (2013) volatile compounds analysis was carried out only in treatments stored at 8 °C, as the volatile compounds emitted by fruit stored at 20 °C would highly increase, masking the ethylene removal effect.

Retention indexes of a commercial alkane standard mixture (Sigma-Aldrich, Steinheim, Germany) were used to identify the compounds, as well as the NIST 17 Mass Spectral and Retention Index Libraries. The identification was considered tentative when it was based only on mass spectral data, and only compounds with spectra similarity >90% were considered as correct hits. The linear retention similarity filter was set at ± 10 units. This volatile compound extraction method has been previously used for the analysis of different food matrices (Noguera-Artiaga et al., 2019, 2020; Pérez-Marín et al., 2021).

2.5. Descriptive sensory analysis

A trained panel consisting of 12 highly-trained panellists (aged 26–55 years; 7 female and 5 male) from the Food Quality and Safety research group (Universidad Miguel Hernández de Elche, UMH, Orihuela, Spain) conducted the descriptive sensory analysis. Each panellist

had more than 800 h of experience with different types of food products, mainly vegetable products. The methodology used for the descriptive sensory analysis was that previously described by Noguera-Artiaga et al. (2019) and Pérez-Marín et al. (2021) The scale used ranged from 10 (extremely high intensity) to 0 (no intensity) with 0.5 increments. Samples were served in odour-free, disposable 100 mL biodegradable cups at room temperature (~22 °C) and were coded using 3-digit numbers. Mineral water and unsalted crackers were provided to panellists to clean their palates between samples. Analyses were run in triplicate (n = 3). Analyses of this parameter were carried out on days 0, 10, 15 and 20. The descriptive sensory analysis focused exclusively on the treatments stored at 8 °C, as we considered the loss of organoleptic quality in the treatments stored at 20 °C to be proven (Massa, Chase, Santini, & Mitchell, 2015).

Participants gave informed consent via the statement "I am aware that my responses are confidential, and I agree to participate in this survey" where an affirmative reply was required to enter the survey. They were able to withdraw from the survey at any time without giving a reason. The products tested were safe for consumption.

The study was conducted according to the guidelines of the Declaration of Helsinki and approved by the Miguel Hernández University of Elche Ethics Committee in 2021. The ethical approval reference number for this study is PRL.DTA.ESN.03.21.

2.6. Statistical analysis

The descriptive statistics (mean and standard error of the mean [SEM]) and the different tests described below were performed using the StatGraphics Centurion XV software (StatPoint Technologies, Inc. Warrenton, VA USA). The Shapiro-Wilk test was performed to check the normality of the data. In addition, to check the homogeneity of variance, Bartlett's test was applied. The data were analysed using an analysis of variance (Two-way ANOVA), as five independent treatments and two factors were available (days 3, 10 and 15). Next, the data were processed using an analysis of variance (One-way ANOVA) when the three independent treatments were available (days 20 and 25) and for all figures. Sensory analysis and volatile composition were analysed using an analysis of variance (One-way ANOVA) comparing the different days studied (0, 10, 15 and 20). Finally, Tukey's Multiple Range Test was utilized to separate means and detect significant differences (p-value < 0.05).

3. Results and discussion

3.1. Physicochemical analysis

3.1.1. SSC

During the post-harvest ripening process in climacteric fruit such as tomatoes, sugars tend to replace acids through certain metabolic processes, giving the fruit a sweet taste and increasing SSC values. (Fig. 1A and Table 1SM).

Statistically, temperature had an influence in the short term, on day 3 ($p < 0.05$). On the other hand, the ethylene factor had an effect in the medium term, on day 15 ($p < 0.001$). No effect of the combination of the two factors were observed.

In treatments kept at 8 °C, SSC increased more smoothly than in treatments kept at 20 °C. In the C treatment the SSC increased by 39.0% from the initial day to day 25, from 4.1 to 5.7. On the CF and CFUv treatments, no differences were observed between days 0 and 25; therefore, the use of the complete system in the CFUv treatment did not affect this parameter. The use of ethylene scavengers in the CF and CFUv treatments resulted in a SSC that was 29.8% lower than the control ($p < 0.001$) on day 25. The effect of ethylene scavengers was more pronounced in treatments kept at 20 °C due to the fact that this temperature is stressful for the fruit, accentuating the effects and the production of ethylene. In the R treatment, SSC increased by 41.5% up to day 15 as compared with the initial day, while the RF treatment remained at the initial levels through the entire storage time (Fig. 1A and Table 1SM).

These changes are related to hydrolytic changes in starch concentration during ripening in the post-harvest period. In tomatoes, the conversion of starch into sugar is an important index of ripening. Mujtaba et al. (2014) showed that ethylene removal in tomato favoured the preservation of SSC. On the other hand, Wills and Ku (2002) found no significant differences in the application of 1-MCP (a synthetic molecule that competes with ethylene for its receptors in fruit) to the preservation of "Clarion" tomatoes compared to the control.

3.1.2. pH

pH is an important factor that can be used to measure the amount of free acids in any fruit, indicating the degradation of organic acids. This parameter is related to SSC, and as the ripening process progresses, sugars replace acids, causing an increase in pH values (Fig. 1B and Table 1SM) (Zhang, Shao, Wei, Xu, & Wang, 2020).

The temperature factor was decisive in the short term on day 3 ($p < 0.01$). On the other hand, the ethylene factor had a greater effect in the medium term on days 10 ($p < 0.001$) and 15 ($p < 0.05$). An interaction of both factors was observed on day 10 ($p < 0.05$).

pH values generally increased throughout the storage time, being affected by temperature and the presence of ethylene scavengers. An 8.6% increase in pH was observed in the C treatment, from 4.44 on the initial day, to 4.82 on day 25. The CF and CFUv treatments showed a pH increase of 5.6%, and no differences were observed ($p > 0.05$) between these two treatments. The effect of the complete system on this parameter was not observed when comparing treatments CF and CFUv. In the R treatment, an increase in pH levels of 6.8% was observed after 15 d of storage, while in the RF treatment, pH increased by 3.9% (Fig. 1B and Table 1SM). The use of ethylene scavengers delayed the increase in pH values in tomatoes preserved at 8 °C and 20 °C.

The effect of ethylene scavengers on pH observed in the above data is in line with results from the literature. Kostekli et al. (2016) reported a deterioration of acids due to tomato ripening processes. As a result, these authors observed an increase in pH levels, being more pronounced in those with higher ethylene concentration. This effect was also observed by Álvarez-Hernández, Martínez-Hernández, Avalos-Belmontes, Miranda-Molina, and Artés-Hernández (2020) in apricot preserved at 15 °C with ethylene removal by $KMnO_4$ oxidation, where differences up to 9% were observed between those preserved with or without ethylene removal. Park, Kim, and Shin (2016) concluded that by applying other ethylene removal methods such as 1-MCP, no significant differences were observed in the pH of the tomatoes tested.

3.1.3. Acidity

Titratable acidity (TA) is a parameter that represents the total amount of acids in the fruit, and is expressed as malic acid equivalents (Fig. 1C and Table 1SM).

Statistically, ethylene factor had an effect on the data on days 3 ($p < 0.05$), 10 ($p < 0.001$), and 15 ($p < 0.001$); on the other hand, the temperature factor had a higher impact on day 3 ($p < 0.001$), and the same on days 10 ($p < 0.001$) and 15 ($p < 0.001$). The combination of both factors was relevant on days 3 ($p < 0.01$), 10 ($p < 0.001$), and 15 ($p < 0.001$).

This parameter was highly affected by the use of ethylene scavengers and the storage temperature. In the control treatment (C), a progressive decline was observed, from 18.8 g L^{-1} to 11.8 g L^{-1} of acids, with a decrease of 37.0%. In contrast, in the CF and CFUv treatments, a moderate decrease was observed, from 18.8 g L^{-1} to 16.9 g L^{-1} in the CF treatment, and from 18.8 g L^{-1} to 17.6 g L^{-1} in the CFUv treatment (a decrease of 10.3% and 6.2%, respectively). These results suggest that the use of ethylene scavengers prevented a drop in TA of 43.2% using $KMnO_4$ filters, and 49.2% aided by the use of the complete system. On the other hand, a more pronounced drop was observed in the treatments kept at 20 °C. From 18.8 g L^{-1} to 9.5 g L^{-1} on day 15 in the R treatment,

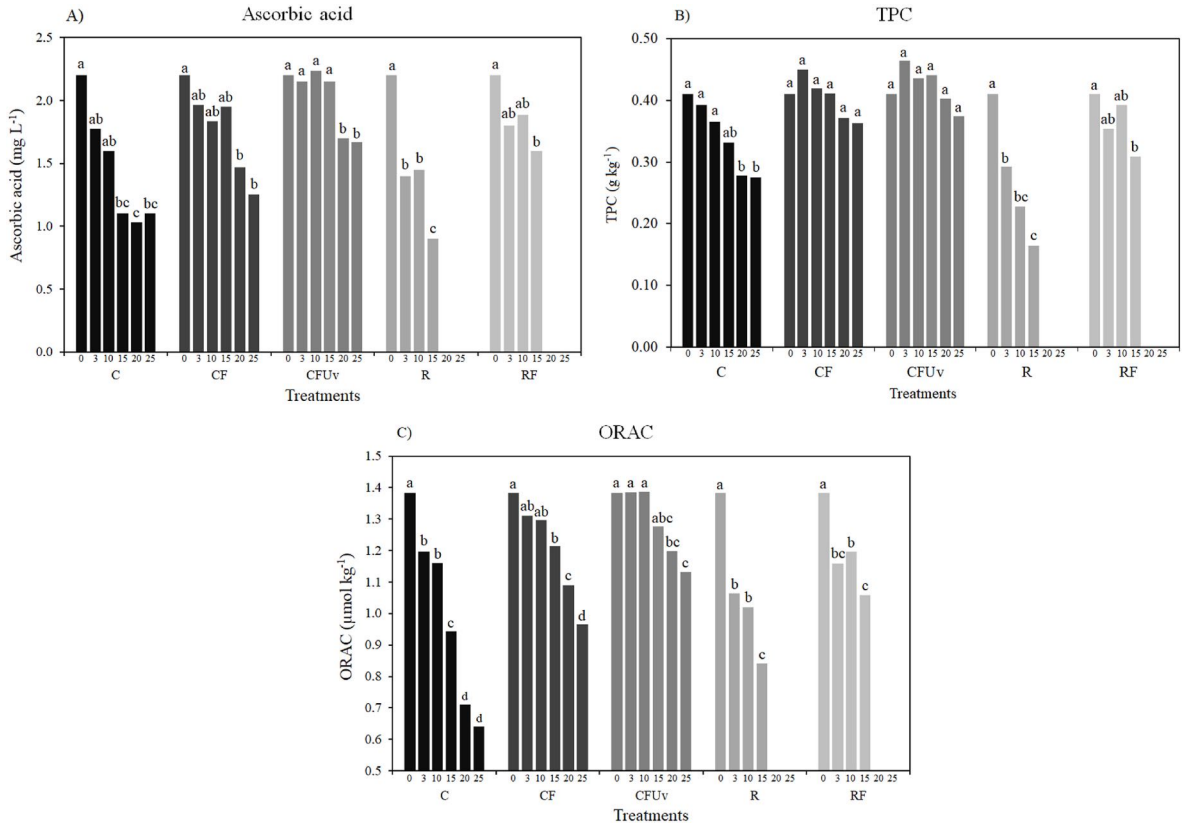

Fig. 2. Evolution during storage time of the antioxidant activity in tomatoes subjected to different treatments (C, CF, CFUv, R and RF). The parameters measured were ascorbic acid content expressed as (mg L^{-1}); ORAC expressed as (µmol kg^{-1}) and total phenolic compounds (TPC) expressed as (g kg^{-1}). Different letters for each treatment represent statistically significant differences according to Tukey's test with the aim to see the evolution of each parameter on every treatment.

and from 18.8 g L^{-1} to 13.7 g L^{-1} in the RF treatment on day 15 (a drop of 49.5% and 27.2%, respectively). In these treatments, the use of ethylene scavengers prevented the decrease in acidity by 60.7% (Fig. 1C and Table 1SM).

Salamanca et al. (2014) already observed a delay in the acid degradation process using $KMnO_4$ as an ethylene scavenger on tomato "Chonto" storage at 18 °C. Mujtaba et al. (2014) also observed this phenomenon on tomato "Rio Grandi", as acids are consumed in the sugar production processes of ripening. This effect increases SSC and pH, and reduces acidity. Thanks to the elimination of ethylene, ripening was delayed and this process was largely avoided. Wills and Ku (2002), observed this effect in tomato using 1-MCP but to a lesser extent, registering a delay of up to 25% in TA after 14 days of storage. Park et al. (2016) observed a similar effect in tomato too.

3.1.4. Ripening index

The ripening index (RI) depends on TA and SSC. This measurement represents the progress in the ripening process of the fruit in numerical form (Fig. 1D and Table 1SM) (Zhang et al., 2020).

Statistically, temperature had an influence throughout the storage period, on day 3 ($p < 0.01$), on day 10 ($p < 0.01$), and on day 15 ($p < 0.001$). On the other hand, the ethylene factor had an effect on days 10 ($p < 0.01$) and 15 ($p < 0.001$). The combination of both factors had an effect on days 10 ($p < 0.001$) and 15 ($p < 0.05$).

The use of ethylene scavengers, and the storage temperature, had a very noticeable effect on this parameter. In the C treatment, a rapid increase was recorded on day 15, from 2.2 to 3.8, which was maintained on day 20, finally reaching its maximum peak on day 25, with a value of 4.9, two-fold higher than the initial day. In the CF treatment, a smooth increase was observed, from 2.2 on the initial day to 2.5 on day 25, two-fold lower than the C treatment on the same day. The CFUv treatment showed an improvement over the other treatments, with no differences from the initial value until day 25. Thus, it maintained the initial ripening level for at least 20 days. On the other hand, in the treatments kept at 20 °C, large differences were recorded. In these treatments, the factor of storage temperature had a great effect. Under these stressful conditions, the fruit ripening and ethylene production processes are accelerated. The increase in RI in the R treatment up to day 15 was three-fold higher, from 2.2 on d 0, to 6.2 on day 15. However, this large increase was not observed in the RF treatment, which increased by 22.7% up to day 15, as compared to the initial measurement (from 2.2, day 0, to 2.7, day 15).

This indicates that the $KMnO_4$ filters were able to delay the increase in RI three-fold. Álvarez-Hernández et al. (2020) observed the same effect in apricots, where ethylene removal resulted in a 60% reduction in RI. Wills and Ku (2002) recorded an RI reduction of 16% by applying 1-MCP to 'Clarion' tomatoes stored for 14 days at 20 °C.

3.1.5. Ascorbic acid

The ascorbic acid content shows the degree of internal degradation of the fruit. This parameter was highly affected by the storage temperature and by the use of ethylene scavengers. The ascorbic acid content tends to degrade over time (Fig. 2A and Table 2SM) (Kostekli et al., 2016). The reason for the degradation lies in processes associated to maturation. This phenomenon leads to tissue rupture and various disorders that result in the liberation of oxidising agents. In order to prevent the degradation caused by these oxidising substances, ascorbic acid reduces their effect through its degradation in the process. Therefore, a higher amount of ascorbic acid in the fruit could be related to a slower ripening (Janjarasskul & Suppakul, 2018).

The statistical analysis by factors for the ascorbic acid analysis (ethylene, temperature, and combination of both) suggested that

temperature had an influence in the short and long terms, on days 3 ($p < 0.001$) and on day 15 ($p < 0.001$). On the other hand, the ethylene factor had an effect throughout the storage period (3, $p < 0.001$, 10, $p < 0.05$, and 15 $p < 0.001$). The combination of both factors had an effect on days 3 ($p < 0.01$) and 15 ($p < 0.001$) (Fig. 2A Table 2SM).

In the C treatment, a progressive decrease was observed, from 2.20 mg L^{-1} on the initial day, to 1.10 mg L^{-1} on day 25. The total loss of ascorbic acid content was 50% for this treatment. However, in the CF and CFUv treatments, the variation observed over the 25 d of the trial was a degradation of ascorbic acid of 43.2% and 25.1%, respectively. These results suggest that the use of $KMnO_4$ filters resulted in a delay in ascorbic acid degradation. In contrast, the use of the complete system ($KMnO_4$ filters, UV light) had a greater effect on this parameter, perhaps due to a more efficient removal of ethylene.

In the R treatment, the decrease was greater than in the treatments kept at 8 °C, from 2.20 mg L^{-1} on day 0, to 0.90 mg L^{-1} on day 15, a drop of 59.1%. However, in the RF treatment, the decrease was less pronounced, from 2.20 mg L^{-1} to 1.60 mg L^{-1} on day 15 (Fig. 2A and Table 2SM). It should be noted that although the RF treatment was stored at 20 °C, it maintained higher ascorbic acid concentration levels than the control treatment. This suggests that ethylene removal by $KMnO_4$ filters may be more important than temperature in the preservation of this parameter.

The data provided suggest that the use of ethylene scavengers strongly affects the amount of ascorbic acid present in tomatoes. According to Lee and Kader (2000), ascorbic acid of tomatoes decreased during storage, supporting the general trend observed in this paper. Also, Mansourbahmani et al. (2018) concluded that 1-MCP caused a delay in ascorbic acid losses of tomatoes in a similar way as the use of $KMnO_4$ and palladium. However, Kostekli et al. (2016) concluded that all the samples of five tomato varieties treated with $KMnO_4$ as ethylene absorber showed a higher content in ascorbic acid content at the end of the storage than the samples without ethylene absorbers.

Table 1
Identification of volatile compounds in tomato samples by HS-SPME.

1.	#	Compound	Retention time (min)	Kovats Index	
				Experimental	Literature
2.	1	2-Hexenal, (E)-	3.511	842	845
3.	2	1-Hexanol	3.624	851	855
4.	3	1-nitro-pentane	4.155	895	900
5.	4	2-Heptenal, (E)-	5.072	946	946
6.	5	1-Octen-3-one	5.442	966	967
7.	6	5-Hepten-2-one, 6-methyl-	5.600	974	979
8.	7	2-amylfuran (Furan, 2-pentyl-)	5.780	984	982
9.	8	2,4-Heptadienal, (E, E)-	5.911	991	998
10.	9	Octanal	6.043	998	1005
11.	10	1-Hexanol, 2-ethyl-	6.624	1020	1026
12.	11	2-Isobutylthiazole	6.921	1031	1038
13.	12	1,4-dibromopentane	7.319	1045	1047
14.	13	2-Decyne	7.405	1048	1050
15.	14	2-Octenal, (E)-	7.485	1051	1044
16.	15	Acetophenone	7.792	1062	1062
17.	16	Perillene	8.734	1097	1099
18.	17	Nonanal	8.877	1101	1102
19.	18	2-Nonenal, (E)-	10.778	1156	1160
20.	19	*cis*-4-Decenal	12.001	1191	1193
21.	20	Decanal	12.478	1204	1205
22.	21	2,4-Nonadienal, (E, E)-	12.833	1213	1215
23.	22	β-Cyclocitral	13.077	1219	1223
24.	23	Citral (Z)	13.704	1235	1240
25.	24	2-Hexenal, (E)-	14.859	1265	1271
26.	25	2,4-Decadienal, (E, E)-	15.957	1292	1300
27.	26	Geranyl acetone	22.181	1445	1450

3.1.6. Phenolic compounds

The results observed in Fig. 2B and Table 2SM show how temperature and the use of ethylene scavengers affected total phenolic compounds (TPC).

Statistically, temperature had an influence throughout the assay, on days 3 ($p < 0.01$), 10 ($p < 0.001$), and 15 ($p < 0.01$). Ethylene had an effect throughout the entire assay on days 3 ($p < 0.05$), 10 ($p < 0.001$), and 15 ($p < 0.05$). As for the combination of the two factors, an effect was observed on days 3 ($p < 0.05$), 10 ($p < 0.01$) and 15 ($p < 0.05$).

In the C treatment, a decrease was observed from day 0, with 0.410 g kg^{-1}, until day 25 with 0.276 g kg^{-1}, a loss of 32.6% of its total phenolic compounds over the storage time. In the CF and CFUv treatments, the decrease was slower, with values remaining close to 0.400 g kg^{-1} throughout the study (Fig. 2B and Table 2SM).

In the treatments stored at 20 °C, the variation of total phenolic compounds was higher. The R treatment showed a reduction from 0.410 g kg^{-1} on day 0, to 0.164 g kg^{-1} by day 15, a drop of 60.0%. In contrast, the RF treatment showed a decrease of 24.8% when comparing the results from day 0 to day 15, decreasing from 0.410 g kg^{-1} to 0.308 g kg^{-1} (Fig. 2B and Table 2SM).

According to (Kader, 2011), the loss of phenolic compounds associated with the advancement of ripening-related processes can result in browning of the tissue, which is undesirable for the quality of appearance. Lopes et al. (2020) stated that maturation is another extremely important factor that can influence the quality of the composition of fruits and vegetables. During the maturation of fruits, several biochemical, physiological and structural modifications occur, affecting the content of health-related phytochemicals like phenolic compounds. Anton et al. (2017) concluded that during tree ripening, tomatoes accumulate a large amount of phenolic compounds until they reach their optimum ripeness. Once they are harvested, it begins a progressive loss of polyphenols and phenolic compounds in general associated with the darkening of the skin. This data agrees with what was found in this study, since in tomatoes stored with ethylene scavengers, a colour gain was observed in the sensory analysis, related to the greater presence of phenolic compounds.

Ethylene removal applied to total phenolic compounds preservation has not been extensively studied. Mansourbahmani et al. (2018) observed that the application of various ethylene removal methods improved the preservation of phenolic compounds. Among them, the use of 20% $KMnO_4$ and 5% palladium resulted in the preservation of up to 40% of the phenolic compounds.

3.1.7. ORAC

In the results obtained from the antioxidant capacity measured with the ORAC method (Fig. 2C and Table 2SM), two different behaviours can be observed in the treatments preserved at 8 °C, and another two in the treatments preserved at 20 °C, affected by the use of ethylene scavengers.

On the one hand, in the C treatment, a 53.6% decrease in antioxidant capacity was recorded after 25 days of storage, falling from 1.38 μmol kg^{-1} on d 0, to 0.64 μmol kg^{-1} on day 25. On the other hand, in the CF treatment, a decrease of 29.7% was observed between day 0 and 25, dropping from 1.38 μmol kg^{-1} to 0.97 μmol kg^{-1} respectively. However, the CFUv treatment decreased from 1.38 μmol kg^{-1} on d 0, to 1.13 μmol kg^{-1} on day 25, which resulted in a 18.1% loss of the initial antioxidant capacity, lower than the other treatments (Fig. 2C and Table 2SM).

As for the treatments stored at 20 °C, the reduction in antioxidant capacity observed in the R treatment after 15 days was 39.1%, while in the RF treatment it was 23.2%. This suggest that the use of ethylene scavengers delayed the loss of antioxidant capacity by 15.9% (Fig. 2C and Table 2SM).

The ethylene factor was statistically determinant throughout the storage time on days 3 ($p < 0.001$), 10 ($p < 0.001$), and 15 ($p < 0.001$), the temperature factor was statistically determinant on days 3 ($p < 0.01$), 10 ($p < 0.001$), and 15 ($p < 0.001$), and the combination of these

Table 2
Volatile composition (% relative area) of tomatoes affected by different treatments (C, CF and CFUv).

1.	#	Volatile compound	ANOVA	Day 0	Day 10			Day 15			Day 20		
					C	CF	CFUv	C	CF	CFUv	C	CF	CFUv
2.	1	2-Hexenal, (E)-	**	40.141 bc	34.933 c	26.266 de	25.144 e	33.811 cd	25.066 d	23.004 d	37.457 c	47.224 ab	52.374 a
3.	2	1-Hexanol	n.s.	8.996	1.095	1.088	2.047	1.125	1.454	2.013	4.547	2.373	5.786
4.	3	1-nitro-pentane	***	6.315 b	4.323 c	12.104 a	7.490 b	3.734 c	11.170 a	6.918 b	1.331 d	3.986 c	3.772 c
5.	4	2-Heptenal, (E)-	***	7.242 a	3.760 bc	3.267 bc	4.273 bc	4.020 bc	3.706 bc	4.458 bc	5.715 ab	4.584 abc	3.042 c
6.	5	1-Octen-3-one	***	0.501 d	1.224 b	0.920 bc	1.585 a	1.038 bc	0.936 bc	1.584 a	0.929 bc	1.119 b	0.694 cd
7.	6	5-Hepten-2-one, 6-methyl-	***	14.858 d	23.694 bc	26.273 abc	21.843 c	29.267 a	27.647 ab	22.637 bc	15.209 d	21.977 c	14.097 d
8.	7	2-amylfuran (Furan, 2-pentyl-)	***	1.073 e	4.283 b	3.272 bc	6.437 a	2.348 cde	2.643 cd	6.524 a	1.704 ed	2.115 cde	2.088 cde
9.	8	2,4-Heptadienal, (E,E)-	***	0.778 d	0.860 cd	1.107 abcd	0.848 cd	1.272 ab	1.398 a	0.932 bcd	0.885 bcd	1.440 a	1.175 abc
10.	9	Octanal	***	0.769 abc	0.987 abc	0.887 abc	1.113 abc	0.691 abc	1.219 ab	1.272 a	0.503 c	0.622 bc	0.569 c
11.	10	1-Hexanol, 2-ethyl-	n.s.	0.143	0.370	0.444	0.107	0.337	0.087	0.086	0.055	0.070	0.027
12.	11	2-Isobutylthiazole	***	3.501 cde	4.042 bc	5.769 a	4.617 b	2.893 e	4.724 b	4.366 bc	1.521 f	3.891 bcd	3.031 de
13.	12	1,4-dibromopentane	**	0.336 cd	0.634 a	0.543 ab	0.636 a	0.468 abc	0.504 abc	0.603 a	0.228 d	0.411 bcd	0.268 d
14.	13	2-Decyne	***	2.153 a	0.451 b	0.349 b	0.347 b	0.470 b	0.507 b	0.325 b	0.841 ab	0.747 ab	0.493 b
15.	14	2-Octenal, (E)-	***	2.713 e	7.509 b	5.822 bcd	10.923 a	6.986 b	6.446 bc	11.578 a	4.706 d	5.195 cd	7.415 a
16.	15	Acetophenone	***	0.277 a	0.221 ab	0.134 abc	0.182 abc	0.182 abc	0.218 ab	0.197 abc	0.065 c	0.106 bc	0.134 abc
17.	16	Perillene	***	0.172 cd	0.408 a	0.358 ab	0.406 a	0.223 bcd	0.272 abc	0.420 a	0.115 d	0.206 bcd	0.126 cd
18.	17	Nonanal	**	1.696 a	1.631 a	1.204 ab	1.551 ab	1.521 ab	1.867 a	1.784 a	0.640 b	0.644 b	0.607 b
19.	18	2-Nonenal, (E)-	***	0.260 de	0.374 b	0.313 bcd	0.517 a	0.297 cde	0.382 b	0.583 a	0.138 f	0.341 bc	0.228 e
20.	19	*cis*-4-Decenal	***	0.280 ab	0.284 ab	0.504 ab	0.410 ab	0.149 b	0.408 ab	0.417 ab	0.671 a	0.266 ab	0.572 ab
21.	20	Decanal	***	0.376 a	0.270 b	0.207 bcd	0.265 bc	0.162 d	0.216 bcd	0.291 ab	0.165 d	0.173 dc	0.204 bcd
22.	21	2,4-Nonadienal, (E,E)-	*	0.025 b	0.136 ab	0.151 a	0.155 a	0.081 bc	0.141 ab	0.166 a	0.015 c	0.044 c	0.036 c
23.	22	β-Cyclocitral	***	0.256 de	0.384 bc	0.394 bc	0.447 ab	0.322 cd	0.372 bc	0.481 a	0.206 d	0.339 c	0.205 d
24.	23	Citral (Z)	***	0.184 d	0.439 bc	0.589 a	0.487 abc	0.548 abc	0.605 a	0.562 ab	0.118 d	0.436 c	0.211 d
25.	24	Geranial (E)	***	1.433 ab	1.258 abc	1.387 ab	0.960 cd	1.515 a	1.531 a	1.093 bcd	1.001 cd	1.541 a	0.827 d
26.	25	2,4-Decadienal, (E,E)-	*	0.273 e	0.974 bc	0.885 bc	1.571 a	0.823 bc	1.006 bc	1.733 a	0.431 de	1.138 b	0.747 cd
27.	26	Geranyl acetone	***	5.248 d	5.457 cd	5.762 cd	5.638 cd	5.714 cd	5.475 cd	5.975 c	5.887 c	6.559 b	8.643 a

Levels of statistical significance are: $*p < 0.05$, $**p < 0.01$ and $***p < 0.001$. n.s: no significant differences. Values (mean of 3 replications) followed by the same letter, within the same volatile descriptor, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test.

factors was relevant though the assay on days 3 ($p < 0.05$), 10 ($p < 0.05$), and 15 ($p < 0.05$).

Therefore, the presence of ethylene and its removal are key factors in the maintenance of the antioxidant capacity of tomato fruit. According to Mansourbahmani et al. (2018) ethylene removal led to the maintenance of antioxidant capacity at each sampling time during storage relative to the control fruit. Also, Álvarez-Hernández et al. (2018) and Kostekli et al. (2016) pointed out that the use of ethylene absorber sachets and storage time had an influence on the antioxidant capacity (ORAC values) of tomato fruit analysed, maintaining them closer to the initial values. Park et al. (2016), using 1-MCP as ethylene blocker concluded that there were no significant differences in antioxidant capacity among treatments on day 15 of storage, indicating that 1-MCP have no effect on the antioxidant contents of tomatoes at the end of storage.

Comparing the results shown in Fig. 2 and Table 2SM, a relationship between the three parameters (ascorbic acid, total phenols and ORAC) can be observed. This is in agreement with the existing literature (Delva & Goodrich-Schneider, 2013). The free radicals released during ripening are stopped by the fruit's own antioxidant agents. The results suggest that the use of ethylene scavengers delays the loss of compounds related to antioxidant capacity.

3.2. Volatile compounds

Twenty-six compounds were found in the analysis of the volatile compounds of tomato samples (Table 1). 2-Hexenal was the most common (~38%), followed by 5-Hepten-2-one, 6-methyl- (~20%), and 2-Octenal (~8%). These compounds are normally predominant in most tomatoes in the early stages of ripening (Alonso, García-Aliaga, García-Martínez, Ruiz, & Carbonell-Barrachina, 2009; Alonso, Vázquez, Garía-Martínez, Ruiz, & Carbonell-Barrachina, 2009; Carbonell-Barrachina, Agustí, & Ruiz, 2006; Pardo-García, Martínez-Gil, López-Córcoles, Zalacain, & Salinas, 2013).

Differences were found in 24 of the 26 compounds found. The 2 compounds that were not affected by storage time and ethylene control systems were 1-Hexanol and 2-Ethyl-1-hexanol.

Trans-2-Hexenal brings freshness to a broad range of fruit and vegetables. It is essential for apple, peach, red fruit, plum, and tomato flavours. As can be observed in Table 2, when the ripening stage of the fruit advances, differences are observed between the ethylene control methods and the control sample. Tomato samples preserved by in the CF and CFUv treatments had higher amounts (% relative area) of *trans*-2-Hexenal than the control sample. This can lead to a higher perception of fresh aroma by the consumer. *Trans*-2-Hexenal compound has been shown to be one of the volatile organic compounds that contributes to the perception of the characteristic tomato aroma (Buttery, 1993). This compound is perceived with sensory notes of "green", "fresh", even "sweet" and among all the volatile organic compounds present in this fruit, it has been found that greater amounts of this compound are related to fresher tomato (Alonso, Vázquez, et al., 2009). Also, it is one of the most important compounds in other fruit, such as apple, melon or kiwifruit (Frank et al., 2007). In addition, it is one of the compounds that undergoes rapid degradation during fruit storage (Wang, Baldwin, Yu, & Bai, 2015). The same occurred with the compound 2-Isobutylthiazole. This compound had higher intensities in the samples preserved by the ethylene control techniques than in the control sample. This compound is sensorially related to fresh tomato descriptors, and is widely used in the production of tomato-based products to provide the products with higher intensities of tomato-ID. This is similar to that observed with 2-Octenal, although for this volatile compound, it was observed that the CFUv treatment stood out for presenting higher intensities. This compound is directly related to green and fresh aromas. This was repeated with other aromatic compounds that are directly related to floral and fresh sensory perceptions, such as Citral, Geranial, Geranyl acetone, or Octanal (Table 2).

The results obtained showed that in general, the ethylene control treatments used managed to maintain the ripening of tomato fruit for a longer period of time. This is reflected in the longer presence of compounds related to green fruit, such as 2-Hexenal, 2-Octenal, 2-Isobutylthiazole, Citral, Geranial, Geranyl acetone, or Octanal (Birtic, Ginies, Causse, Renard, & Page, 2009; Tobaruela et al., 2021).

Table 3
Descriptive sensory analysis of tomatoes affected by different treatments (C, CF and CFUv).

1. Sensory descriptor	ANOVA	Day 0	Day 10			Day 15			Day 20		
			C	CF	CFUv	C	CF	CFUv	C	CF	CFUv
2. **Appearance**											
3. Colour	***	6.50 d	7,15 c	6.85 cd	7.10 c	8.15 b	8.00 b	7.37 c	9.05 a	8.60 a	8.05 b
4. Colour homogeneity	***	5.45 d	6.80 c	7.00 c	6.90 c	7.85 ab	8.15 ab	7.65 ab	9.30 a	8.95 a	9.10 a
5. Shine	**	4.50 a	2.80 b	3.10 b	2.85 b	3.00 b	2.50 b	2.75 b	1.45 c	1.66 c	1.55 c
6. Flavour											
7. Sweet	**	2.25 d	2.65 c	3.10 b	2.50 c	3.25 ab	3.85 ab	2.65 bc	4.50 a	3.85 b	3.50 b
8. Sour	n.s.	3.05	2.10	2.60	2.85	2.25	3.00	3.00	2.15	2.55	2.66
9. Tomato ID	**	5.15 a	4.55 b	4.40 b	5.10 a	3.25 c	4.37 b	5.05 a	2.20 d	3.85 c	4.50 b
10. Fruity	n.s.	1.55	2.05	2.56	2.45	3.25	3.75	2.50	3.15	3.20	2.95
11. Vegetal	**	6.52 a	5.60 b	5.80 b	5.40 b	4.25 c	4.37 c	5.15 b	3.60 d	3.05 d	4.45 c
12. Aftertaste	**	5.55 a	5.35 a	5.50 a	5.50 a	4.25 b	4.75 b	3.75 c	2.35 d	2.10 d	3.46 c
13. Texture											
14. Hardness	***	7.25 a	6.25 b	6.45 b	6.55 b	4.75 c	5.85 c	5.75 c	2.25 d	2.85 d	2.85 d
15. Crunchiness	**	6.75 a	5.85 b	5.60 b	5.76 b	5.50 b	5.35 bc	4.85 c	2.50 d	3.25 d	3.10 d
16. Juiciness	*	4.80 e	5.10 d	5.45 d	5.80 d	6.87 c	6.75 c	7.57 b	7.25 b	7.25 b	8.30 a
17. Density of juice	n.s.	2.25	2.25	2.40	2.10	2.25	2.00	2.25	1.80	1.75	1.85
18. Pulp amount	*	6.58 a	6.25 a	6.30 a	6.15 a	5.85 ab	6.15 a	4.65 bc	5.05 b	5.20 b	4.15 c
19. Skin amount	n.s.	2.2	2.30	2.15	2.16	1.88	1.63	1.63	2.20	1.95	2.05
20. Seeds and juice	n.s.	2.5	2.15	2.35	2.60	2.25	2.50	3.75	2.15	2.25	2.33
21. Saliva solubility	n.s.	4.32	4.50	4.65	4.90	5.37	5.25	5.35	5.25	5.36	5.55
22. Residual skin	n.s.	4.15	4.05	4.20	4.15	4.13	4.00	4.13	4.50	4.05	4.20

Levels of statistical significance are: $^{*}p < 0.05$, $^{**}p < 0.01$ and $^{***}p < 0.001$. n.s: no significant differences. Values (mean of 3 replications) followed by the same letter, within the same sensory descriptor, were not significantly different ($p > 0.05$), according to Tukey's least significant difference test.

3.3. Descriptive sensory analysis

After the descriptive sensory analysis of the tomato samples, differences were found in 11 of the 18 sensory descriptors studied (Table 3).

The colour of the tomato was affected by the different conservation methods from the second sampling day (day 15). The samples preserved by in the CFUv treatment showed a less intense colour evolution than the C and CF samples. In other words, this system managed to preserve the green-red colour of the tomato for a longer period of time. A similar effect was observed with the Sweetness, Tomato-ID, Vegetal, and Aftertaste descriptors; the samples preserved with the CFUv treatment showed less variations during the storage time of the tomatoes, with respect to the C and CF samples.

In the case of texture (Hardness & Crunchiness), no differences were observed between the samples, for the same preservation time, with the different methods used. Variations were observed in Juiciness and Pulp-amount sensory descriptors; the samples preserved in the CFUv treatment had higher Juiciness and lower Pulp-amount than those preserved with the C and CF treatments.

It was observed that throughout the ripening process, the homogeneity and brightness of the fruit were uniformly modified; differences were observed between the different storage times but not between the different preservation methods.

These sensory results allow us to conclude that the CFUv treatment manages to reduce the ripening of the fruit for a longer period of time, preserving the colour, the tomato flavour, the vegetable aftertaste, and the texture of the tomato. No effects were found using the CF treatment.

4. Conclusions

The results obtained provide clear evidence that the use of $KMnO_4$ filters favoured a better preservation of the parameters observed in the CF and RF treatments with respect to the C treatment preserved at 8 °C, and the R treatment storage at 20 °C. However, the use of the combination of potassium permanganate with UV-C radiation (CFUv treatment) had a greater effect maintaining a low SSC level, pH, and RI; it also had an important effect on the preservation of ascorbic acid and antioxidant capacity (ORAC), as compared to C and CF treatments. The presence of volatile compounds related to early ripening stages in fruit (such as 2-Hexenal, 2-Octenal, 2-Isobutylthiazole, Citral, Geranial, Geranyl acetone, or Octanal) was also observed in the CFUv treatment as compared to other refrigerated treatments (C and CF). Differences were found in 11 of the 18 compounds evaluated in the descriptive sensory analysis, the CFUv treatment scored better than the other treatments, with the maintenance of low levels of ripening, especially in the descriptors of colour, flavour, vegetal aftertaste, and texture. The data shown suggest that the fruit preserved with the $KMnO_4$ and UV-irradiation (CFUv) system were less ripe than the rest, with a longer shelf life.

CRediT authorship contribution statement

Ramiro Alonso-Salinas: Conceptualization, Data curation, Formal analysis, Investigation, Software, Visualization, Writing – original draft, Writing – review & editing. **Santiago López-Miranda:** Conceptualization, Formal analysis, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – review & editing. **Antonio J. Pérez-López:** Conceptualization, Data curation, Methodology, Supervision, Validation, Writing – original draft, Writing – review & editing. **Luis Noguera-Artiaga:** Data curation, Methodology, Writing – original draft. **Ángel A. Carbonell-Barrachina:** Methodology, Resources, Supervision. **Estrella Núñez-Delicado:** Funding acquisition, Investigation, Project administration, Resources, Writing – review & editing. **José Ramón Acosta-Motos:** Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – review & editing.

Declaration of competing interest

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Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary data

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