# AGRICULTURAL AND FOOD CHEMISTRY

## 1-Methylcyclopropene Increases Storability and Shelf Life in Climacteric and Nonclimacteric Plums

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The effect of 1-methylcyclopropene (1-MCP) at three different doses (0.25, 0.50, and 0.75  $\mu$ L L<sup>-1</sup>) on the ripening processes of a climacteric, cv. Santa Rosa, and a suppressed climacteric type, cv. Golden Japan, plum was studied. For both cultivars, positive effects were observed in terms of inhibition of ethylene production and delays of the physical, chemical, and biochemical changes associated with ripening. 1-MCP-treated plums were firmer with lower weight loss, reduced °Brix/titratable acidity ratios, and lower color changes during cold storage and subsequent shelf life at 20 °C than controls. For most factors, the effectiveness of 1-MCP was dose-dependent in Santa Rosa but dose-independent for Golden Japan.

#### KEYWORDS: 1-MCP; ethylene; firmness; ripening index; plum ripening; postharvest storage

#### INTRODUCTION

The plum has been categorized as climacteric fruit, with increases in both ethylene and respiration rate during ripening. However, this is not a general behavior, since some cultivars such as Golden Japan, Shiro, and Rubyred show a suppressed climacteric pattern, while Santa Rosa, Gulfruby, and BlackStar behave as typical climacteric fruits (1-3). Plums are highly perishable and require special care during handling and storage, although suppressed climacteric cultivars have longer shelf lives than climacteric ones (4).

1-Methylcyclopropene (1-MCP) is a nontoxic antagonist of the ethylene hormone that binds to the ethylene receptor after treatment (5). 1-MCP has been extensively used on fruits at the experimental level in order to understand the mechanisms controlling the physiology and biochemistry of the ripening processes, and the effect of 1-MCP in delaying ripening has been studied on climacteric and nonclimacteric fruits (6, 7). In most of these studies, the treatments were performed at ambient temperature, and most of the times, only delays at the onset of the ethylene production were achieved, but total inhibition was not observed. 1-MCP is commercially used in cut flowers and apples while its use in other fruits and vegetables is still being investigated. The aim of this work was to study the effect of 1-MCP in two plum cultivars: Golden Japan, which is considered as a nonclimacteric phenotype, and Santa Rosa, which behaves as a climacteric fruit. 1-MCP treatments at

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different doses (0.25, 0.50, and 0.75  $\mu$ L L<sup>-1</sup>) and at low temperature in already cooled fruits were performed, and ethylene production, respiration rate, firmness, color, and °Brix/ titratable acidity ratio were analyzed during cold storage and further during shelf life. Also, the ethylene dependence on these parameters and the possible application of 1-MCP on delaying the ripening process and increasing storability and shelf life of plums are discussed.

#### MATERIAL AND METHODS

Plant Material. Plum fruits (Prunus salicina Lindl., cv. Golden Japan and cv. Santa Rosa) were harvested at the commercial ripening stage from 10 year old trees grown in an orchard in Murcia (Spain). For both cultivars, over 3500 fruits were manually picked, to avoid mechanical damage, and once in the laboratory, they were selected according to size and color, randomized, and divided into 147 lots of 20 plums. Three lots were used to analyze the fruit properties at harvest (day 0). Four groups of 36 lots were used for 1-MCP treatments in triplicate: 0 (control), 0.25, 0.50, and 0.75  $\mu$ L L<sup>-1</sup> (see 1-MCP Treatment). After treatments, plums were stored at 1  $\pm$  0.5 °C, in permanent darkness and with a relative humidity of 90%. The schedule of sampling was as follows: 1 day after treatment and then weekly for 5 weeks. Six lots were sampled for each treatment; three lots were analyzed immediately, and three lots were stored at 20 °C for 4 days to evaluate shelf life and to determine ethylene, respiration rate, weight loss, color, firmness, and °Brix/titratable acidity ratio.

**1-MCP Treatment**. Fruits were precooled at 1 °C for 6 h before treatments. SmartFresh (0.14%) was supplied by AgroFresh Inc. (Rohm and Haas Inc., Gessate, Italy) as a powder, which after warm water (40 °C) was added, released 1-MCP as a gas. Different amounts of the powder (in triplicate) were weighed, and suitable amounts of warm water were added to obtain stock solutions of 1-MCP in a sealed glass

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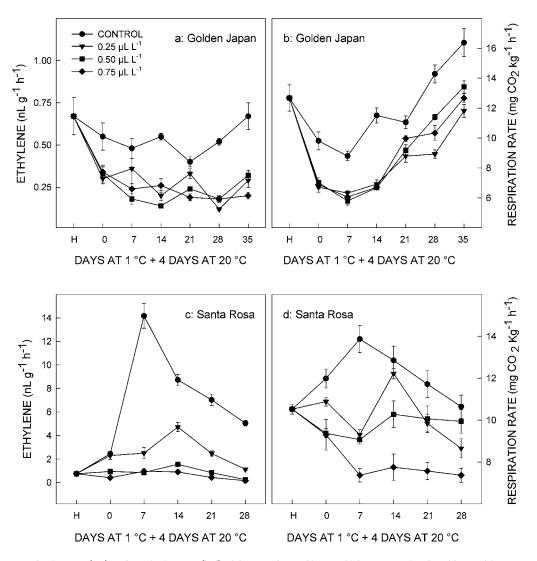


Figure 1. Ethylene production rate (a,c) and respiration rate (b,d) of the two plum cultivars, which were previously cold stored for 0–5 weeks, at harvest (H) and after 4 days at 20 °C.

jar, from which 50 mL was taken and injected into 120 L hermetically sealed containers to reach concentrations of 0.25, 0.50, and 0.75  $\mu$ L L<sup>-1</sup>. The duration of treatment was 24 h at 1 °C. Control fruits were treated similarly but without 1-MCP.

Ethylene and Respiration Rate Determination. Ethylene and CO<sub>2</sub> production were measured on 20 fruit replicates in 3 L glass jars hermetically sealed with a rubber stopper for 1 h. One milliliter of the holder atmosphere was withdrawn with a gas syringe, and the ethylene was quantified using a Hewlett-Packard model 5890A gas chromatograph (Wilmington, DE) equipped with a flame ionization detector and a 3 m stainless steel column with an inner diameter of 3.5 mm containing activated alumina of 80/100 mesh. The column temperature was 90 °C, and the injector and detector temperatures were 150 °C. Results were the mean of four determinations for each replicate and expressed as nL  $g^{-1}$  h<sup>-1</sup>. For respiration rate determination, another sample of 1 mL of the same atmosphere was withdrawn and CO2 was quantified using a Shimadzu 14A gas chromatograph (Kyoto, Japan), with a thermal conductivity detector and a molecular sieve 5A column, 80-100 mesh (Carbosieve SII, Supelco Inc., Bellefonte, U.S.A.), of 2 m length and 3 mm i.d. The oven and injector temperatures were 50 and 110 °C, respectively. Helium was used as a carrier gas at a flow rate of 50 mL min<sup>-1</sup>. Results were the mean of four determinations for each replicate and expressed as mg CO<sub>2</sub> kg<sup>-1</sup> h<sup>-1</sup>.

**Color.** Two color determinations were made on each fruit using the Hunter Lab System (L, a, b) in a Minolta colorimeter CR200 model (Minolta Camera Co., Osaka, Japan) and expressed as L parameter and

chroma index [chroma =  $(a^2 + b^2)^{1/2}$ ]. Results were the mean of triplicate lots of 20 fruits.

**Flesh Firmness.** For each fruit,  $1 \text{ cm}^2$  of the skin was removed and the Magness—Taylor force (flesh fruit firmness indicator) measurement was individually recorded using a 5 mm diameter probe coupled with a TX-XT2i Texture Analyzer (Stable Microsystems, Godalming, U.K.) interfaced to a personal computer. A beveled holder prevented bruising of the opposite side. The penetration rate was 20 mm min<sup>-1</sup> for 10 mm after contacting the flesh, and results were the mean of triplicate lots of 20 fruits and expressed in N.

°Brix/Titratable Acidity Ratio. The total soluble solids concentration (°Brix) was determined in each fruit with a digital refractometer Atago PR-101 (Atago Co. Ltd., Tokyo, Japan) at 20 °C. The titratable acidity (g of malic acid equivalent per 100 g<sup>-1</sup> fresh weight) was determined in each fruit by potentiometric titration with 0.1 N NaOH up to pH 8.1, using 1 mL of diluted juice in 25 mL of distilled H<sub>2</sub>O.

**Statistical Analysis.** Data were subjected to analysis of variance. Sources of variation were storage duration and treatments. Mean comparisons were performed using HSD Tukey's test to examine if differences between treatments and storage duration were significant at  $P \le 0.05$ . All analyses were performed with SPSS software package v. 11.0 for Windows. Linear regressions were performed between 1-MCP applied dose and each parameter taking into account all sampling data, either during cold storage or after 4 days of shelf life at 20 °C.

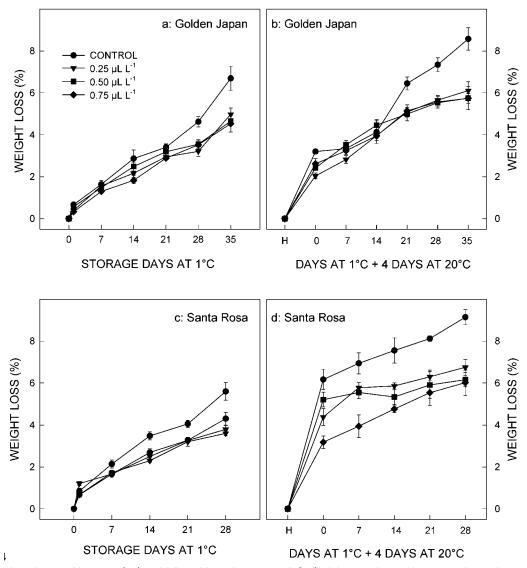


Figure 2. Weight loss during cold storage (a,c) and followed by 4 days at 20 °C (b,d) of the two plum cultivars. H is data at harvest.

### **RESULTS AND DISCUSSION**

Ethylene Production and Respiration Rate. Ethylene production is decreased by cold storage but increased when fruit are transferred to ambient temperature. Ethylene production of both cultivars was less than 0.5 nL  $g^{-1} h^{-1}$  during cold storage, and no significant differences between control and 1-MCPtreated fruits were detectable (data not shown). When fruits were transferred to 20 °C after cold storage, the Golden Japan cultivar still exhibited a low ethylene production rate, but control plums showed higher levels ( $P \le 0.05$ ) as compared with 1-MCPtreated ones (Figure 1a). The same effect was observed for the Santa Rosa cultivar, although a peak of ethylene production was shown in control plums (14.17  $\pm$  1.05 nL g<sup>-1</sup> h<sup>-1</sup>) after 1 week of cold storage plus 4 days at 20 °C (Figure 1c). Inhibition (P  $\leq$  0.05) of ethylene production was observed for 1-MCP-treated plums, although in the 0.25  $\mu$ L L<sup>-1</sup> 1-MCP treatment a slight and delayed ethylene production peak was shown after 14 days of cold storage plus 4 days at 20 °C (4.72  $\pm$  0.40 nL g<sup>-1</sup> h<sup>-1</sup>). Results from linear regressions revealed that ethylene production was dose-dependent during the shelf life for Santa Rosa (y = $-7.75x + 5.62, r^2 = 0.84$ ).

Thus, the effectiveness of the treatment on the inhibition of ethylene emission was clear, especially for the climacteric cultivar Santa Rosa at 0.50 and 0.75  $\mu$ L L<sup>-1</sup> doses (**Figure 1c**).

However, in other climacteric fruits, such as Beauty, Gulfruby, and Royal Zee plum cultivars (4, 8), Canino apricots (8), Cavendish bananas (9), and Hakuho peaches (10), after treatment with distinct 1-MCP doses (most of them higher than those used in the present study), the onset of ethylene production was delayed rather than totally inhibited. In these reports, the 1-MCP treatments were performed at 20 °C or a higher temperature, while in the present work they were carried out at 1 °C on plums and the ethylene production was totally inhibited. In suppressed climacteric plum types, which produce very low levels of ethylene ( $\cong 0.5 \text{ nL g}^{-1} \text{ h}^{-1}$ ), the inhibition of ethylene after 1-MCP treatment is achieved independently of the temperature at which the treatment was performed, as has been observed in Red Rosa, Rubyred, and Shiro plums treated at 20 °C (4, 11) and for Golden Japan treated at 1 °C (Figure 1a).

No differences (P < 0.05) in respiration rate were found between control and 1-MCP-treated plums of either cultivar during cold storage (data not shown). During the shelf life after cold storage, the respiration rate of Golden Japan plums was inhibited in 1-MCP-treated fruit as compared with control ones, irrespective of the 1-MCP dose (**Figure 1b**). In Santa Rosa, control and 0.25  $\mu$ L L<sup>-1</sup> 1-MCP-treated plums exhibited a respiration climacteric peak coinciding with the ethylene one (13.86 ± 0.64 and 12.22 ± 0.25 mg CO<sub>2</sub> kg<sup>-1</sup> h<sup>-1</sup>, respectively),

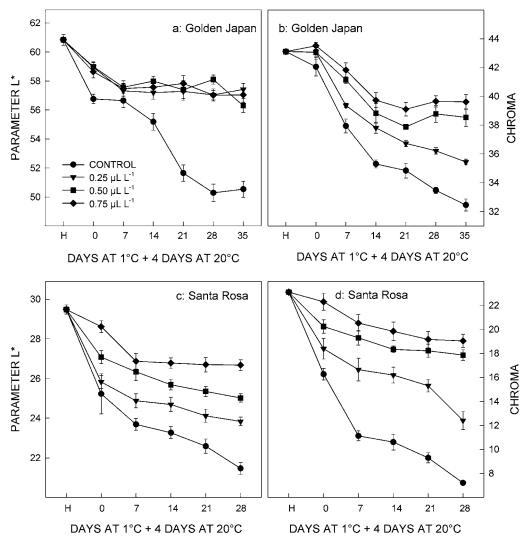


Figure 3. Color L\* (a,c) and chroma index (b,d) of the two plum cultivars, which were previously cold stored for 0–5 weeks, at harvest (H) and after 4 days at 20 °C.

while for 0.5 and 0.75  $\mu$ L L<sup>-1</sup> 1-MCP-treated plums, respiration rate was inhibited ( $P \le 0.05$ ) and no climacteric respiration peak was detected (**Figure 1d**). Similar results have been reported for other fruits, confirming the inhibitory role of 1-MCP in the respiration rate (8-10).

Weight Loss. The loss of weight during storage is an important cause of fruit quality deterioration. Weight loss increased during both cold storage and shelf life of the plums but was greater ( $P \le 0.05$ ) in control than in 1-MCP-treated plums (Figure 2). Fan et al. (12), however, did not find differences between control and treated apricot with higher 1-MCP doses (1  $\mu$ L L<sup>-1</sup>) than used in this work. The higher weight loss could be related to the higher respiration rate found in control fruit with respect to treated plums (Figure 1b,d). From these results, it can be inferred that weight loss is not ethylenedependent, since similar amounts of weight losses were obtained for both cultivars, but Santa Rosa had a higher ethylene production than Golden Japan. In addition, no significant linear regressions between weight loss and 1-MCP applied doses for both cultivars, either during cold storage or shelf life, were detected.

**Fruit Parameters Related to Ripening.** For both cultivars, color did not change during cold storage, and no differences ( $P \le 0.05$ ) were detected between control and 1-MCP-treated plums (data not shown). However, in Golden Japan,  $L^*$  and

chroma decreased during shelf life (**Figure 3a,b**); these changes were greater ( $P \le 0.05$ ) in control than in 1-MCP-treated plums. Significant differences were also found among doses for chroma index (**Figure 3b**). An inverse correlation was detected between chroma index and 1-MCP applied dose (y = 4.29x + 24.33,  $r^2 = 0.99$ ). In Santa Rosa, both  $L^*$  and chroma decreased at 20 °C; the decrease was higher ( $P \le 0.05$ ) in control than in 1-MCP-treated fruits (**Figure 3c,d**), in which the magnitude of the effect of 1-MCP on delaying the decrease in both parameters was dose-dependent (y = 2.68x + 24.98,  $r^2 = 0.98$  for parameter  $L^*$  and y = 10.23x + 13.67,  $r^2 = 0.94$  for chroma index).

At harvest, Santa Rosa fruits were firmer  $(9.70 \pm 0.04 \text{ N})$  than Golden Japan fruits  $(6.62 \pm 0.46 \text{ N})$ . Both cultivars softened during storage but to a greater extend in control than in 1-MCP-treated plums (**Figure 4**). At the end of cold storage, control plums showed flesh firmness levels of  $1.70 \pm 0.11 \text{ N}$  in Golden Japan (**Figure 4a**) and  $4.61 \pm 0.27 \text{ N}$  in Santa Rosa (**Figure 4c**). During the shelf life period after cold storage, the softening process still continued, and the loss of firmness was higher in control than in 1-MCP-treated plums, especially for Santa Rosa (**Figure 4b,d**). High correlation was detected between levels of flesh firmness and 1-MCP applied dose in Golden Japan during cold storage (y = 1.44x + 4.04,  $r^2 = 0.74$ ) and shelf life (y = 0.84x + 2.29,  $r^2 = 0.65$ ), as well as in Santa

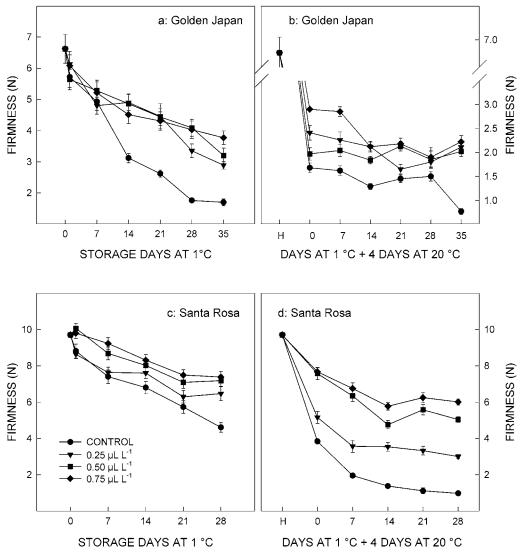


Figure 4. Flesh firmness (Magness–Taylor force) during cold storage (a,c) and followed by 4 days at 20 °C (b,d) of the two plum cultivars. H is data at harvest.

Rosa during cold storage (y = 1.71x + 7.69,  $r^2 = 0.94$ ) or further shelf life (y = 5.05x + 3.78,  $r^2 = 0.95$ ).

The ratio between °Brix and titratable acidity increased to a greater extent during cold storage and further shelf life in control than in 1-MCP-treated plums for both plum cultivars (**Figure 5**). In Golden Japan, the evolution of °Brix/titratable acidity ratio was not correlated to the 1-MCP applied dose. In Santa Rosa, the reduction of the increase in the °Brix/titratable acidity ratio was highly correlated to the 1-MCP applied dose, both during cold storage (y = -0.68x + 7.45,  $r^2 = 0.79$ ) and further shelf life (y = -3.48x + 9.24,  $r^2 = 0.96$ ).

Fruit softening is a ripening processes that is sensitive to ethylene (13, 14). Consumers associate fruit quality with an optimum level of firmness together with desirable aroma, flavor, and color. In this work, increased °Brix/titratable acidity ratio, color changes, and loss of firmness occurred rapidly in control plums, during both cold storage and shelf life (**Figures 3–5**). In contrast, 1-MCP-treated plums maintained higher firmness levels and had less color variation and lower increases in the °Brix/titratable acidity ratio than control fruits. These results show that 1-MCP was able to delay the normal changes that occurr during the postharvest ripening processes, which could be considered as a general effect of 1-MCP treatment, since similar effects have been reported in apricot, banana, avocado,

and other plum cultivars (8, 12, 14, 15). The effectiveness of 1-MCP was, in general, more accentuated in Santa Rosa than in Golden Japan, and 1-MCP effects for most of the analyzed parameters during cold storage and further shelf life in Santa Rosa were dose-dependent. In the suppressed climacteric Golden Japan, 1-MCP delayed the changes related to fruit quality but not in a dose-dependent manner. The 0.25  $\mu$ L L<sup>-1</sup> of 1-MCP was enough to reach the desirable effects on delaying the evolution of ripening parameters. Thus, the optimal dose of 1-MCP to achieve the desirable effects would be higher for those plum cultivars with greater ethylene production during the climacteric stage. In terms of shelf life, it could be established that in control plums the maximum storage period at 1 °C with optimum quality parameters is 2 weeks for both cultivars, while in 1-MCP-treated plums this period is increased up to 5 weeks for Golden Japan and 4 weeks for Santa Rosa. When control fruits were transferred to 20 °C, both cultivars overripened and were unmarketable after 1 week of cold storage and 4 days at 20 °C. Those fruits treated with 1-MCP showed good properties for consumption even after 4 weeks of cold storage plus 4 days at 20 °C for both Golden Japan and Santa Rosa cultivars.

In conclusion, 1-MCP could be a useful tool to prolong the storability of plum fruit since ethylene production was drastically inhibited and the biochemical changes associated with ripening

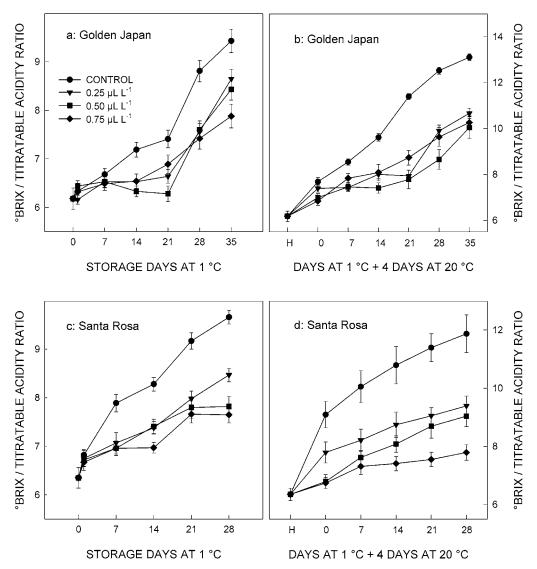


Figure 5. °Brix/titratable acidity ratio during cold storage (a,c) and followed by 4 days at 20 °C (b,d) of the two plum cultivars. H is data at harvest.

were significantly delayed. In Santa Rosa, the majority of these changes was 1-MCP dose-dependent; efficiency was probably linked to ethylene production rate. In the suppressed climacteric Golden Japan, all 1-MCP doses showed similar results. Therefore, for commercial purposes, the 1-MCP concentration required to get the desirable effects will be a function of the normal ethylene production rate of each cultivar.

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#### LITERATURE CITED

- Abdi, N.; Holford, P.; McGlasson, W. B.; Mizrahi, Y. Ripening behaviour and responses to propylene in four cultivars of Japanese type plums. *Postharvest Biol. Technol.* **1997**, *12*, 21– 34.
- (2) Zuzunaga, M.; Serrano, M.; Martínez-Romero, D.; Valero, D.; Riquelme, F. Comparative study of two plum (*Prunus salicina* Lindl.) cultivars during growth and ripening. *Food Sci. Technol. Int.* **2001**, *7*, 123–130.

- (3) Pérez-Vicente, A.; Martínez-Romero, D.; Carbonell, A.; Serrano, M.; Riquelme, F.; Guillén, F.; Valero, D. Role of polyamines in extending shelf life and the reduction of mechanical damage during plum (*Prunus salicina* Lindl.) storage. *Postharvest Biol. Technol.* 2002, 25, 25–32.
- (4) Abdi, N.; McGlasson, W. B.; Holford, P.; Williams, M.; Mizrahi, Y. Responses of climacteric and suppressed-climacteric plums to treatment with propylene and 1-methylcycloprpopene. *Postharvest Biol. Technol.* **1998**, *14*, 29–39.
- (5) Sisler, E. C.; Serek, M.; Dupille, E. Comparison of cyclopropene, 1-methylcyclopropene, and 3,3-dimethylcyclopropene as ethylene antagonists in plants. *Plant Growth Regul.* **1995**, *17*, 1–6.
- (6) Blankenship, S. M.; Dole, J. M. 1-Methylcyclopropene: a review. Postharvest Biol. Technol. 2003, 28, 1–25.
- (7) Watkins, C. B. Ethylene synthesis, mode of action, consequences and control. In *Fruit Quality and Its Biological Basis*; Knee, M., Ed.; Academic Press: Sheffield, 2002.
- (8) Dong, L.; Lurie, S.; Zhou, H. W. Effect of 1-methylcyclopropene on ripening of Canino apricots and Royal Zee plums. *Postharvest Biol. Technol.* 2002, 24, 135–145.
- (9) Jiang, Y.; Joyce, D. C.; Macnish, A. J. Extension of the shelf life of banana fruit by 1-methylcyclopropene in combination with polyethylene bags. *Postharvest Biol. Technol.* **1999**, *16*, 187– 193.

- (10) Mathooko, F. M.; Tsunashima, Y.; Owino, W. Z. O.; Kubo, Y.; Inaba, A. Regulation of genes encoding ethylene biosynthetic enzymes in peach (*Prunus persica* L.) fruit by carbon dioxide and 1-methylcyclopropene. *Postharvest Biol. Technol.* 2001, 24, 265–281.
- (11) Dong, L.; Zhou, H. W.; Sonego, L.; Lers, A.; Lurie, S. Ripening of Red Rosa plums: effect of ethylene and 1-methylcyclopropene. *Aust. J. Plant Physiol.* **2001**, *28*, 1039–1045.
- (12) Fan, X.; Argenta, L.; Mattheis, J. P. Inhibition of ethylene action by 1-methylcyclopropene prolongs storage life of apricots. *Postharvest Biol. Technol.* **2000**, *20*, 135–142.
- (13) Lelièvre, J. M.; Latché, A.; Jones, B.; Bouzayen, M.; Pech, J. C. Ethylene and fruit ripening. *Physiol. Plant.* **1997**, *101*, 727–739.

- (14) Golding, J. B.; Shearer, D.; Wyllie, S. G.; McGlasson, W. B. Application of 1-MCP and propylene to identify ethylene dependent ripening processes in mature banana fruit. *Postharvest Biol. Technol.* **1998**, *14*, 87–98.
- (15) Jeong, J.; Huber, D. J.; Sargent, S. A. Influence of 1-methylcyclopropene (1-MCP) on ripening and cell-wall matrix polysaccharides of avocado (*Persea americana*) fruit. *Postharvest Biol. Technol.* 2002, 25, 241–2561.

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