

Article

Effects of Preharvest and Postharvest Salicylic Acid and Oxalic Acid Treatment on the Long Cold Storage Quality of ‘Lane Late’ Navel Oranges and ‘Summerina’ Mandarins

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Abstract

Preharvest and postharvest treatments are widely used to maintain citrus fruit quality during extended cold storage. This study evaluated the effects of pre- and postharvest applications of salicylic acid (SA) and oxalic acid (OA), applied separately and in combination, on the quality of ‘Lane Late’ navel oranges and ‘Summerina’ mandarins at harvest and stored at 1 °C for 10 weeks followed by 1 week at 20 °C. Fruit quality attributes, including weight loss, fungal decay, firmness, total soluble solids (TSS), titratable acidity (TA), electrolyte leakage, and biochemical markers, were assessed. Overall, treatments had limited and inconsistent effects on most quality parameters, with no significant differences observed in chilling injury, respiration rate, ethylene production, ethanol content, vitamin C content, malondialdehyde content, or colour. At harvest, oranges subjected to preharvest OA treatments had lower TSS and TA, while peel antioxidant capacity (AOC) was lower for oranges treated with preharvest SA or OA. After storage, postharvest SA reduced calyx stem-end rot by 30% in mandarins. Electrolyte leakage was reduced by preharvest SA in oranges and postharvest OA in mandarins, indicating improved membrane stability. No consistent synergistic effects were observed for combined treatments, and responses varied between cultivars, highlighting the importance of cultivar-specific optimisation. These findings indicate that SA and OA provide limited but targeted benefits under extended cold storage conditions.



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1. Introduction

Citrus (*Citrus* L.) is one of the most widely cultivated fruit crops globally, where final fruit quality is influenced by cultivar, production system, and postharvest handling practices [1,2]. In Australia, ‘Summerina’ mandarins (*Citrus reticulata*) are a recently developed cultivar, valued for their seedless, easy peeling characteristics and suitability for both domestic and export markets [3]. Similarly, ‘Lane Late’ navel oranges (*Citrus sinensis*) are widely grown due to their extended harvest window and desirable internal quality [4]. De-

spite their commercial relevance, limited information is available regarding the postharvest behaviour and quality retention of these cultivars under extended cold storage conditions.

The Australian citrus industry relies heavily on long-distance supply chains and export markets, making extended cold storage essential for maintaining fruit quality [5]. However, prolonged storage at low temperatures can lead to physiological disorders such as chilling injury (CI), which negatively affects the fruits' appearance and marketability [6–8]. CI symptoms in citrus include peel pitting, flavedo browning, and rind staining, and are associated with disruptions in cellular metabolism integrity [6,8,9]. Susceptibility to CI varies among cultivars and is influenced by many preharvest and postharvest factors [6,10].

To mitigate quality deterioration during long cold storage, various preharvest and postharvest treatments have been investigated [11]. Among these, salicylic acid (SA) is a well-established signalling molecule known to enhance stress tolerance and induce systemic acquired resistance [1]. SA has been reported to reduce respiration rate, delay softening, and maintain fruit quality during storage for mandarins, oranges and lemons, while also alleviating CI through improved antioxidant activity and membrane stabilization [1,12–15]. In citrus, preharvest and postharvest SA treatments have been shown to reduce decay incidence, maintain firmness, and preserve quality attributes such as TSS and TA [12,16,17]. While SA has been well-studied in citrus, limited information is available comparing preharvest and postharvest applications under long cold storage.

Oxalic acid (OA) has also emerged as a promising postharvest treatment with reported roles in delaying senescence, reducing decay and enhancing antioxidant systems [18]. Preharvest OA has been shown to improve lemon fruit firmness, maintain internal quality and alleviate CI by modulating oxidative stress and antioxidant responses [18,19]. However, compared to SA, fewer studies have examined the effects of OA in citrus, with limited studies conducted during cold storage, and its efficacy may vary depending on species, cultivar and treatment conditions [18,19]. Currently, there are limited reports on the combination effects of SA and OA on citrus during long cold storage.

This study aimed to evaluate the effects of preharvest and postharvest applications of SA and OA, on the quality of 'Lane Late' navel oranges and 'Summerina' mandarins during extended cold storage. Specifically, the study assessed changes in CI, respiration rate, ethylene production, electrolyte leakage, AOC, malondialdehyde content, decay incidence, and key internal and external quality parameters after 10 weeks of cold storage at 1 °C with an additional 1 week at 20 °C shelf life. This study provides insights into the effectiveness of these novel treatments under long cold storage conditions relevant to commercial cold storage and export supply chains [10,11].

2. Materials and Methods

2.1. Treatment and Storage Conditions

'Summerina' mandarin fruit on Trifoliata rootstock and 'Lane Late' navel oranges on Citrange rootstock in a commercially maintained orchard in Mildura, Victoria, Australia, were used in this trial. Five orchard rows were allocated to each treatment for each cultivar, with two buffer rows maintained between adjacent treatment rows to minimize spray drift. For 'Summerina' mandarins, 415 trees were assigned to each treatment, while for 'Lane Late' oranges, 760 trees were assigned to each treatment. Trees were sprayed at 6, 4 and 2 weeks before harvest with either water (control), 5 mM of preharvest oxalic acid (OA) or 4 mM of preharvest salicylic acid (SA) that was dissolved in 1% ethanol [16,18]. The 4 mM SA concentration was selected based on previous studies demonstrating beneficial effects on citrus fruit quality and chilling tolerance during storage [12,16]. The 5 mM OA concentration was selected because this concentration has been shown to be effective in other fruit systems, while reports on citrus are limited [18]. Following harvest, fruits from

treated orchard sections were randomly collected and allocated into four replicate boxes per treatment. Fruits were further dipped with either water (control), 5 mM OA or 4 mM SA, which was dissolved in 1% ethanol then dipped for 5 min [16,18]. A total of nine treatment combinations were evaluated based on preharvest application and postharvest dipping treatments: (1) water only preharvest spray and postharvest dip, untreated; (2) preharvest salicylic acid; (3) preharvest oxalic acid; (4) postharvest salicylic acid dip; (5) postharvest oxalic acid dip; (6) preharvest salicylic acid and postharvest salicylic acid dip; (7) preharvest salicylic acid and postharvest oxalic acid; (8) preharvest oxalic acid and postharvest salicylic acid dip; and (9) preharvest oxalic acid and postharvest oxalic acid dip. Each of the different treatment combinations contained four replicates.

After treatment, all fruits were packed in cardboard boxes and transported for 3 days through the supply chain to the NSW Department of Primary Industries and Regional Development (Ourimbah, NSW, Australia) by refrigerated trucks, followed by storage at 1 ± 1 °C for 10 weeks and for an additional week at ambient temperature (20 ± 1 °C) to allow for chilling injury expression and shelf-life stimulation [13]. Each treatment contained temperature and relative humidity loggers (Tinytag TV-4020 and TV-4500 data loggers, Gemini, Chichester, UK) to monitor the treatment, transport and storage conditions.

2.2. Quality Assessments

2.2.1. Weight Loss and Decay Assessments

Fruit weight loss (%) was calculated by weighing 10 individual fruits upon treatment, then re-weighing the same fruit after 10 weeks of storage at 1 °C plus one week at 20 °C. Fruit with decay were excluded from analysis. Weight loss was calculated as the percentage reduction in fruit weight relative to the initial weight recorded at harvest [20].

The decay (%) was calculated as total decay among replicates compared to total fruit within individual replicates. Fruit was assessed for decay at 10 weeks storage at 1 °C plus one week at 20 °C. Fruit was assessed for postharvest decay (*Penicillium* moulds and stem-end rots). Fruit with any decay were not included in the following quality assessments.

2.2.2. Visual Assessments of Overall Quality

Fruit quality was assessed by visual assessment of all 40 oranges from each replicate and all 80 mandarins from each replicate. The overall acceptability was assessed using a five-point scale where 1 = excellent condition, firm texture, good colour and gloss; 2 = good quality; 3 = acceptable quality; 4 = unacceptable, soft; and 5 = unacceptable, very soft [21]. Fruit was assessed at harvest and at 10 weeks storage at 1 °C plus one week at 20 °C. CI was assessed on both cultivars based on the presence of pitting, necrosis or red blotching symptoms [13]. CI severity was scored using a four-point scale, where 1 = normal (no pitting), 2 = slight pitting (a few scattered pits, up to 5%), 3 = moderate pitting (6% to 30% surface coverage), and 4 = severe pitting (>30% surface coverage) [13,22]. Fruit was assessed at 10 weeks storage at 1 °C plus one week at 20 °C. Calyx quality was assessed on oranges using a five-point scale, where 1 = green (0% yellow/brown), 2 = slightly yellow (1–25% yellow/brown), 3 = moderately yellow (25–50% yellow/brown), 4 = mostly yellow (50–75% yellow/brown), and 5 = brown (>75% yellow/brown) [22,23]. Fruit was assessed at harvest and at 10 weeks storage at 1 °C plus one week at 20 °C.

2.2.3. Fruit Colour

Ten oranges per treatment unit were measured for skin colour (L^* , a^* , b^* values) using a Minolta chroma meter model CR-400 (Minolta Co. Ltd., Osaka, Japan). The colorimeter was calibrated using a white standard calibration plate before each measurement time. Fruit was assessed for colour at harvest and at 10 weeks storage at 1 °C plus one week at 20 °C. The colour of each fruit was measured at two points around the fruit equator,

following standard colorimetric procedures. Hue angle and chroma values were calculated using Equations (1) and (2) [24].

$$\text{Hue angle} = \text{Hue} = \tan^{-1} \left(\frac{b^*}{a^*} \right) \quad (1)$$

$$\text{Chroma} = \sqrt{(a^*)^2 + (b^*)^2} \quad (2)$$

2.2.4. Ethylene Production and Respiration Rate

Ethylene production and respiration rate were measured according to Archer et al. (2021) [25]. Two randomly selected fruit per replicate were sealed in a 1.5 L hermetic glass jar with septum and lid for gas sampling after 2 h. The jars were held at 20 °C for 2 h before a sample of headspace carbon dioxide and ethylene gases was withdrawn to measure the rate of CO₂ and ethylene production. Ethylene measurements were conducted at harvest and after 10 weeks storage at 1 °C plus one week at 20 °C. At harvest, a 1 mL gas sample was taken from the glass jar and inserted into a Felix gas analyser (F-950 three gas analyser, Felix instruments, Camas, WA, USA). Fruit was assessed at 10 weeks' storage at 1 °C plus one week at 20 °C, the headspace sample (1 mL) was measured using a gas chromatograph (Nexis GC-2300, Shimadzu Corporation, Kyoto, Japan). Nitrogen was used as the carrier gas for the gas chromatograph. Ethylene production was expressed as µL C₂H₄ kg⁻¹ h⁻¹ and calculated according to Equation (3).

$$\text{Ethylene production (}\mu\text{L C}_2\text{H}_4 \text{ kg}^{-1}\text{h}^{-1}\text{)} = \frac{\text{C}_2\text{H}_4 \left(\frac{\mu\text{L}}{\text{L}} \right) \times \text{volume of container (L)}}{\text{Initial produce weight(kg)} \times \text{time (h)}} \quad (3)$$

Fruit was assessed at harvest and at 10 weeks' storage at 1 °C plus one week at 20 °C for respiration rate. The respiration rate was determined by measuring CO₂ in 5 mL of gas sample withdrawn from the glass jar and injected into an ICA40 series low-volume gas analysis system (International Controlled Atmosphere Ltd., Kent, UK). Respiration rate was expressed as mL CO₂ kg⁻¹ h⁻¹ and calculated according to Equation (4).

$$\text{Respiration rate (mL CO}_2 \text{ kg}^{-1} \text{ h}^{-1}\text{)} = \frac{\% \text{CO}_2 \times \text{Volume of container (mL)}}{\text{Initial produce weight(kg)} \times 100 \times \text{time (h)}} \quad (4)$$

2.2.5. Fruit Firmness

Fruit firmness was measured at harvest and at 10 weeks storage at 1 °C plus one week at 20 °C, using a texture analyser (TA1, AMETEK Lloyd Instruments Ltd., Fareham, UK) fitted with a 70 mm flat square compression plate with a crosshead speed of 200 mm min⁻¹ with a pre-loaded stress of 0.05 kgf. Each fruit was measured twice, then rotated 90° across the equator to be additionally measured two more times, totalling four measurements per fruit. Fruit firmness was expressed as kilograms force (kgf) [25].

2.2.6. Total Soluble Solids (TSS), Titratable Acidity (TA) and Maturity Index (TSS:TA)

TSS and TA were measured at harvest and at 10 weeks storage at 1 °C plus one week at 20 °C. TSS, expressed as % Brix, was determined by measuring the juice of five fruits per treatment replicate, with a digital refractometer (Atago PAL-1, Tokyo, Japan). TA was determined by titrating 5 mL of freshly prepared juice with 0.1 M NaOH to pH 8.2 using an automatic titrator (Mettler Toledo Titrator Excellence T5, Greifensee, Switzerland), and the results were expressed as percentage of citric acid equivalents [24]. The TSS:TA maturity index was calculated by dividing the measured TSS (% Brix) by the TA (% citric acid) value for each sample.

2.2.7. Vitamin C

Vitamin C content was determined according to Alhassan et al. [22]. Vitamin C was measured at harvest and at 10 weeks storage at 1 °C plus one week at 20 °C. Freshly pre-

pared juice from five fruits of each treatment unit was used to estimate the vitamin C content using the iodometric titration method and expressed as $\mu\text{L L}^{-1}$ ascorbic acid equivalents.

2.2.8. Ethanol

Ethanol concentration was determined using a gas chromatograph (Gow-Mac Series 580, Gow-Mac Instrument Co., Bridgewater, NJ, USA), according to Archer et al. [25]. Ethanol was measured at harvest and after 10 weeks of storage at 1 °C plus one week at 20 °C. Juice samples from five fruits per replicate (10 mL) were transferred into sealed vials and placed in a water bath at 30 °C for 10 min. The headspace gas samples were withdrawn and injected into the gas chromatograph for analysis. The results were expressed as $\mu\text{L L}^{-1}$.

2.2.9. Peel Antioxidant Capacity (AOC)

The peel AOC was determined according to Papoutsis et al. [26], with slight modifications. AOC was measured at harvest and after 10 weeks of storage at 1 °C plus one week at 20 °C. Specifically, 2,2-diphenyl-1-picrylhydrazyl (DPPH) solution was produced by dissolving 24 mg DPPH in 100 mL of methanol and then stored at −20 °C. To prepare the working solution, 10 mL of solution was mixed with 45 mL of methanol to obtain an absorbance of 1.1 ± 0.02 at 515 nm using a Thermo Scientific Genesys 150 UV-Visible Spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). Four and a half mL of methanol was mixed with 0.5 mL sample of peel supernatant. Then, 2.85 mL of working solution was mixed with 125 μL of the previous mixed sample and left in darkness for 3 h before measuring the absorbance at 515 nm [26]. The results were expressed as mg Trolox equivalents per grams of sample fresh weight ($\mu\text{mol TE g}^{-1} \text{FW}$) [26].

2.2.10. Malondialdehyde (MDA) Content

The MDA concentration was measured according to Habibi et al. [27], with slight modifications. MDA was measured at harvest and at 10 weeks storage at 1 °C plus one week at 20 °C. A mixture of 2.5 g of citrus peel samples with 10 mL of trichloroacetic acid (TCA) was centrifuged at 10,000 rpm at 4 °C for 20 min. Then, 2 mL supernatant was mixed with 2 mL thiobarbituric acid (TBA) and heated in a water bath at 95 °C for 20 min, then cooled. The mixture was centrifuged at 10,000 rpm for 5 min at room temperature. After the supernatant was collected, its absorbance was measured at 450 nm, 532 nm and 600 nm using a Thermo Scientific Genesys UV-Visible spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). Peel MDA content was calculated according to the Formula (5).

$$\text{MDA } (\mu\text{mol kg}^{-1} \text{FW}) = \frac{[6.45 \times (A_{532} - A_{600}) - 0.56 \times A_{450}] \times V_t \times V_r}{V_s \times w} \quad (5)$$

V_t , V_r , and V_s represent the total volume of the extract solution, the total volume of the reaction mixture solution, and the volume of the extract solution contained in the reaction mixture solution, respectively, and w represents the weight of the sample. The content of MDA was expressed as $\mu\text{mol kg}^{-1} \text{FW}$.

2.2.11. Electrolyte Leakage (EL)

EL was measured according to Wang et al. [28] with slight modifications. EL was measured at harvest and after 10 weeks of storage at 1 °C plus one week at 20 °C. A total of 25 disks of peel samples of 1 cm diameter were put into 30 mL of Milli-Q-water and shaken for 2.5 h. Initial EL was measured using a conductivity meter (edge HI2003, Hanna Instruments, Woonsocket, RI, USA) after shaking (EC_1). Samples were placed in a freezer for 15 h, then thawed and autoclaved at 120 °C for 15 min. Once back to room temperature, the final electrical conductivity (EC_2) of the solution was measured. Electrolyte leakage was calculated as a percentage using the following Equation (6).

$$EL(\%) = \frac{EC1}{EC2} \times 100 \quad (6)$$

2.3. Statistical Analysis

A one-way analysis of variance (ANOVA) and Tukey's post hoc test were conducted using the IBM SPSS statistical software for Windows, Version 30 (IBM Corp., Armonk, NY, USA). Data are reported as mean values $\pm$ standard error, and differences between the means ($n = 4$) were considered statistically different at $p \leq 0.05$.

3. Results and Discussion

3.1. 'Lane Late' Navel Oranges

3.1.1. Postharvest Quality and Physiological Responses of 'Lane Late' Oranges

There were no significant differences in fruit weight loss (%), decay (%), CI score, calyx score, colour parameters (hue angle and chroma), ethylene production and respiration rate between the treated and the untreated 'Lane Late' navel oranges at either harvest or after 10 weeks at 1 °C followed by 1 week at 20 °C (Table 1). While some differences between the treatments were observed, these treatment differences were inconsistent and not considered a critical factor for this study.

The main citrus postharvest decay was blue mould, caused by *Penicillium italicum*, with lesser decay due to green mould, caused by *Penicillium digitatum*, and brown rot. While postharvest salicylic acid (SA) application has been reported to induce systemic acquired resistance and enhance antifungal defence responses in mandarins during cold storage, the timing of application appears critical [1,12]. In the present study, the lack of significant reduction in decay incidence suggests that neither preharvest nor postharvest SA or oxalic acid (OA) provide sufficient protection under external cold storage conditions.

Oranges treated with preharvest OA followed by postharvest SA had the highest preferred overall acceptability score (2.87). No significant differences were observed between the singular preharvest treatments and singular postharvest treatments compared to the untreated citrus. All treatments were considered to be of acceptable quality range, indicating minimal differences between the treated samples.

These observations show no consistent differences in physiological responses, decay incidence and visual assessments in 'Lane Late' navel fruit quality between the different pre- and postharvest OA and SA treatments during cold storage. Furthermore, the combined application of OA and SA did not result in synergistic effects under the conditions tested. This lack of response may be attributed to the timing or concentration of the treatments. While SA is known to activate stress pathways [1], its effectiveness is often dependent on its application relative to stress exposure, and similar constraints may apply to OA.

While previous studies have reported reductions in weight loss following preharvest OA application in other citrus species (e.g., lemons at 1 mM [19]), no such effect was observed in the present study, suggesting that treatment responses may be cultivar- or peel-structure-dependent [19]. In contrast, Haider et al. [12] reported reduced *Penicillium* decay following SA application, particularly under higher disease pressure or when applied after harvest, highlighting the importance of treatment timing and storage conditions. Similarly, preharvest applications of OA and both pre- and postharvest applications of SA have been reported to alleviate CI in certain citrus cultivars; however, in this study, no reduction in CI symptoms were observed in 'Lane Late' oranges, indicating a limited response under cold storage at 10 weeks [13,19,29,30].

Table 1. Weight loss, decay, chilling injury score, overall acceptability score, calyx score, colour, ethylene production and respiration rate of treated ‘Lane Late’ navel oranges assessed at harvest then stored for 10 weeks at 1 °C plus 1 week at 20 °C.

	Weight Loss (%)	Decay (%)	Chilling Injury Score	Overall Acceptability Score	Calyx Score	Colour (Chroma)	Colour (Hue)	Ethylene Production ($\mu\text{L C}_2\text{H}_4 \text{ kg}^{-1} \text{ h}^{-1}$)	Respiration Rate ($\text{mL CO}_2 \text{ kg}^{-1} \text{ h}^{-1}$)
				Harvest					
Untreated				1.09 $\pm$ 0.03 ^a	1.15 $\pm$ 0.05 ^a	76.56 $\pm$ 0.31 ^a	73.40 $\pm$ 0.41 ^a	11.76 $\pm$ 3.95 ^a	15.97 $\pm$ 1.45 ^a
Preharvest salicylic acid				1.11 $\pm$ 0.04 ^a	1.14 $\pm$ 0.05 ^a	76.86 $\pm$ 0.33 ^a	72.38 $\pm$ 0.53 ^a	15.28 $\pm$ 2.00 ^a	16.48 $\pm$ 0.57 ^a
Preharvest oxalic acid				1.10 $\pm$ 0.03 ^a	1.21 $\pm$ 0.06 ^a	76.52 $\pm$ 0.40 ^a	72.09 $\pm$ 0.52 ^a	12.07 $\pm$ 2.69 ^a	13.55 $\pm$ 1.65 ^a
				10 weeks at 1 °C and 1 week at 20 °C					
Untreated	7.40 $\pm$ 0.17 ^a	12.07 $\pm$ 3.61 ^{ab}	1.58 $\pm$ 0.08 ^{ab}	3.49 $\pm$ 0.06 ^b	3.74 $\pm$ 0.09 ^{ab}	74.54 $\pm$ 0.42 ^{ab}	73.26 $\pm$ 0.34 ^a	0.27 $\pm$ 0.13 ^a	14.05 $\pm$ 0.94 ^a
Preharvest salicylic acid	6.73 $\pm$ 0.15 ^a	17.33 $\pm$ 4.43 ^{ab}	1.75 $\pm$ 0.09 ^{ab}	3.24 $\pm$ 0.09 ^{ab}	3.94 $\pm$ 0.10 ^b	75.21 $\pm$ 0.39 ^{ab}	73.66 $\pm$ 0.31 ^a	0.14 $\pm$ 0.06 ^a	15.20 $\pm$ 1.61 ^a
Preharvest oxalic acid	6.76 $\pm$ 0.15 ^a	24.91 $\pm$ 5.56 ^b	1.75 $\pm$ 0.10 ^{ab}	3.16 $\pm$ 0.10 ^{ab}	3.61 $\pm$ 0.08 ^{ab}	75.21 $\pm$ 0.41 ^{ab}	73.42 $\pm$ 0.37 ^a	0.31 $\pm$ 0.11 ^a	13.61 $\pm$ 0.13 ^a
Postharvest salicylic acid	7.89 $\pm$ 0.20 ^a	20.10 $\pm$ 2.47 ^{ab}	1.54 $\pm$ 0.08 ^a	3.13 $\pm$ 0.08 ^{ab}	3.66 $\pm$ 0.10 ^{ab}	75.31 $\pm$ 0.51 ^{ab}	73.05 $\pm$ 0.38 ^a	0.06 $\pm$ 0.02 ^a	14.63 $\pm$ 0.37 ^a
Postharvest oxalic acid	7.85 $\pm$ 0.14 ^a	14.57 $\pm$ 3.41 ^{ab}	1.55 $\pm$ 0.08 ^a	3.13 $\pm$ 0.09 ^{ab}	3.38 $\pm$ 0.13 ^a	75.90 $\pm$ 0.31 ^{ab}	73.87 $\pm$ 0.30 ^a	0.07 $\pm$ 0.04 ^a	13.49 $\pm$ 0.54 ^a
Preharvest salicylic acid and postharvest salicylic acid	7.71 $\pm$ 0.15 ^a	12.53 $\pm$ 2.80 ^{ab}	1.87 $\pm$ 0.10 ^{ab}	3.19 $\pm$ 0.09 ^{ab}	3.66 $\pm$ 0.10 ^{ab}	74.24 $\pm$ 1.18 ^a	73.07 $\pm$ 0.36 ^a	0.21 $\pm$ 0.10 ^a	13.38 $\pm$ 0.77 ^a
Preharvest salicylic acid and postharvest oxalic acid	7.32 $\pm$ 0.29 ^a	8.54 $\pm$ 2.45 ^a	1.69 $\pm$ 0.09 ^{ab}	3.31 $\pm$ 0.08 ^b	3.89 $\pm$ 0.08 ^b	76.34 $\pm$ 0.50 ^{ab}	73.66 $\pm$ 0.35 ^a	0.11 $\pm$ 0.04 ^a	11.53 $\pm$ 2.27 ^a
Preharvest oxalic acid and postharvest salicylic acid	7.33 $\pm$ 0.43 ^a	11.10 $\pm$ 2.48 ^{ab}	1.50 $\pm$ 0.08 ^a	2.87 $\pm$ 0.07 ^a	3.67 $\pm$ 0.08 ^{ab}	77.06 $\pm$ 0.24 ^b	74.15 $\pm$ 0.32 ^a	0.21 $\pm$ 0.09 ^a	13.63 $\pm$ 1.48 ^a
Preharvest oxalic acid and postharvest oxalic acid	7.15 $\pm$ 0.14 ^a	20.91 $\pm$ 0.98 ^{ab}	1.96 $\pm$ 0.11 ^b	3.22 $\pm$ 0.10 ^{ab}	3.75 $\pm$ 0.10 ^{ab}	76.08 $\pm$ 0.54 ^{ab}	73.87 $\pm$ 0.35 ^a	0.16 $\pm$ 0.05 ^a	13.14 $\pm$ 0.99 ^a

Values are means of four replicates $\pm$ standard error. Different letters in each column indicate significant differences between treatments at each assessment time (Tukey HSD, $p \leq 0.05$). Chilling injury score: 1 = normal (no pitting) and 4 = severe pitting (>30% surface coverage). Overall acceptability score: 1 = excellent condition and 5 = unacceptable, very soft. Calyx score: 1 = green (0% yellow/brown) and 5 = brown (>75% yellow/brown).

Previous studies [1] have suggested that SA may delay chlorophyll degradation and influence peel colour development; however, in this trial the fruit were at the peak of the season and at commercial maturity with full orange colour and no significant changes in hue angle or chroma were detected between treatments. Although SA has been associated with suppression of ethylene production and respiration in some fruit systems, the absence of significant changes in these parameters is consistent with the non-climacteric nature of citrus, which is characterised by low ethylene production and the relatively stable storage conditions applied [1]. Limited information is available regarding the effect of OA or SA on calyx senescence in citrus, and no treatment effects were observed in the present study [22].

3.1.2. Physicochemical and Oxidative Parameters of 'Lane Late' Oranges

For 'Lane Late' navel oranges, there were no significant treatment effects on firmness, vitamin C content, ethanol content, malondialdehyde (MDA) content between treatments at harvest or after 10 weeks at 1 °C followed by 1 week at 20 °C (Table 2). Although minor variations were observed between treatments, these were not generally significantly different or consistent, indicating that the applied SA and OA treatments did not influence physicochemical or oxidative parameters during storage.

At harvest, oranges treated with preharvest OA had lower TSS and TA compared with the untreated fruits and the preharvest SA (Table 2). Despite these differences, the TSS:TA maturity index was not significantly affected at harvest. At 10 weeks of storage, no significant treatment effects were observed for TSS, TA, and the maturity index (TSS:TA). The lower TSS and TA in 'Lane Late' navel oranges at harvest following the preharvest OA applications suggest a potential influence on carbohydrates and organic acid metabolism. However, the lack of change in maturity index (TSS:TA) indicates limited impact on overall flavour balance [31]. Changes in TSS and TA following preharvest treatments have been reported in citrus, although responses are often inconsistent and cultivar-dependent [32,33].

At harvest, untreated oranges exhibited higher peel AOC, compared with fruit treated either with preharvest SA or OA (Table 2). However, these differences were not maintained after storage, while no significant treatment effects were observed at week 10. The lower AOC observed at harvest in treated fruit may reflect a transient alteration in antioxidant metabolism following preharvest SA and OA application. Similar treatments have been reported to enhance antioxidant enzyme activities, including catalase (CAT), peroxidase (POD), and ascorbate peroxidase (APX), in citrus fruit, indicating that changes in AOC may occur during the preharvest period [19,30]. Under stress conditions, increased ROS generation can temporarily exceed AOC, resulting in measurable antioxidant content. Although enzymatic antioxidant activities such as CAT, POD and APX were not measured in the present study, previous studies have reported increases in these enzymes following preharvest SA and OA treatments in citrus fruits. For example, Serna-Escolano et al. [19] reported on an increase in CAT, APX and POD activity in lemons treated during preharvest with 1 mM OA. Similar results have reported by Haider et al. [12] on mandarins treated with 4 mM SA during postharvest. The initial decrease in AOC may reflect a short-term consumption of antioxidant compounds in response to an increase in oxidative stress following treatment and harvest. However, unlike previous reports, the differences in AOC were not observed during cold storage for 10 weeks plus 1 week at ambient temperature [12,34]. These results suggest that the initial changes in antioxidant metabolism were not maintained over cold storage.

MDA is a marker of lipid peroxidation and oxidative stress, and in this trial, its stability across the different treatments suggests that oxidative damage was not significantly altered by SA or OA applications [6,9]. The absence of change in antioxidant activity during storage suggests that these treatments did not enhance the fruit's general antioxidant defence

systems during storage. These findings contrast with studies reporting antioxidant activity increased following SA or OA application, again highlighting the influence of experimental conditions, including treatment concentration, time, and storage environment. Lower MDA accumulation following SA or OA treatments has been reported in citrus, suggesting reduced lipid peroxidation and oxidative damage [19,35,36].

In this study, EL was lower in oranges treated with preharvest SA, as compared with untreated oranges (Table 2). This may indicate improved membrane stability in response to SA applications, although this effect was not reflected by other oxidative stress indicators. This response may reflect improved membrane stability in SA-treated fruit, which is closely associated with oxidative stress regulation and antioxidant activity. Reductions in EL following SA treatments have been associated with improved membrane stability and reduced cellular damage [37].

To evaluate whether treatment responses were dependent on citrus type, SA and OA treatments were applied to ‘Summerina’ mandarins (Tables 3 and 4).

3.2. ‘Summerina’ Mandarins

3.2.1. Postharvest Quality and Physiological Responses of ‘Summerina’ Mandarins

There were no significant differences observed between the different treatments for weight loss (%), overall acceptability, CI scores, ethylene production and respiration rate in ‘Summerina’ mandarins at harvest or after 10 weeks at 1 °C followed by 1 week at 20 °C (Table 3). Although minor variations were observed among treatments, these were not considered relevant to this study. These observations are consistent with those observed in ‘Lane Late’ oranges, indicating that SA and OA treatments did not influence key physiological parameters under the storage conditions evaluated.

In ‘Summerina’ mandarins, the predominant form of decay in mandarins was stem-end rot (Table 3). Stem-end rot represents a major infection pathway in mandarins, as the calyx region provides a natural entry point for the pathogens *Lasiodiplodia theobromae* and *Phomopsis citri* during postharvest handling and storage [38]. The untreated mandarins exhibited the highest stem-end rot incidence (58%), whereas postharvest treatments reduced decay, with fruit treated with postharvest SA showing the lowest incidence (28%). Combination treatments involving preharvest SA also reduced stem-end rot compared to the untreated mandarins. The improved control of decay following SA application may be attributed to infection sites and surface microflora, as well as to its role in activating defence-related pathways, including systemic acquired resistance and antioxidant systems [1,17]. Similar reductions in decay following SA treatments have been reported in mandarins and other citrus cultivars, supporting the role of postharvest SA in enhancing disease resistance [17,39]. Such variability in treatment response between citrus types has been widely reported and is often attributed to differences in peel morphology and infection pathways [40,41].

Minor differences in skin colour (hue value) were observed at harvest (Table 3). Pre-harvest SA ‘Summerina’ mandarin fruit exhibited a slight shift towards more yellow hue compared with the control; however, the colour difference was small and was not reflected in visual assessments. Similarly, no clear treatment effects were detected for overall acceptability, indicating that fruit quality was largely maintained irrespective of treatment.

Overall, while OA and SA applications had minimal impact on physiological and quality attributes, postharvest SA treatments were effective in reducing stem-end rot in ‘Summerina’ mandarins. This observation contrasts with the lack of decay control observed in ‘Lane Late’ oranges, suggesting a cultivar-dependent response to pre- and postharvest treatments. The greater efficacy of postharvest treatment in mandarins may reflect differences in peel structure, infection pathways or susceptibility to calyx-related decay, where direct surface treatment is more effective in suppressing pathogen development.

Table 2. Firmness, titratable acidity (TA), total soluble solids (TSS), the maturity index (TSS:TA), vitamin C, ethanol content, peel antioxidant capacity (AOC), malondialdehyde content (MDA) and electrolyte leakage (EL) of treated ‘Lane Late’ navel oranges assessed at harvest then stored for 10 weeks at 1 °C plus 1 week at 20 °C.

	Firmness (kgf)	TA (% Citric Acid)	TSS (% Brix)	TSS:TA	Vitamin C ($\mu\text{L L}^{-1}$)	Ethanol ($\mu\text{L L}^{-1}$)	AOC ($\mu\text{mol TE g}^{-1}\text{ FW}$)	MDA ($\mu\text{mol kg}^{-1}\text{ FW}$)	EL (%)
Harvest									
Untreated	8.31 $\pm$ 0.19 ^a	0.87 $\pm$ 0.01 ^b	12.33 $\pm$ 0.22 ^a	14.23 $\pm$ 0.12 ^a	522.50 $\pm$ 10.31 ^a	299.22 $\pm$ 94.01 ^a	528.87 $\pm$ 14.29 ^b	31.58 $\pm$ 2.43 ^a	35.65 $\pm$ 0.31 ^a
Preharvest salicylic acid	7.87 $\pm$ 0.26 ^a	0.81 $\pm$ 0.04 ^{ab}	12.23 $\pm$ 0.09 ^a	15.24 $\pm$ 0.68 ^a	542.50 $\pm$ 24.96 ^a	164.99 $\pm$ 23.96 ^a	375.89 $\pm$ 10.10 ^a	21.07 $\pm$ 0.67 ^a	32.79 $\pm$ 1.09 ^a
Preharvest oxalic acid	8.37 $\pm$ 0.29 ^a	0.74 $\pm$ 0.01 ^a	10.78 $\pm$ 0.09 ^b	14.56 $\pm$ 0.21 ^a	532.50 $\pm$ 19.74 ^a	465.40 $\pm$ 344.46 ^a	389.53 $\pm$ 18.90 ^a	29.87 $\pm$ 3.51 ^a	35.55 $\pm$ 0.56 ^a
10 weeks at 1 °C and 1 week at 20 °C									
Untreated	5.06 $\pm$ 0.10 ^{abc}	0.69 $\pm$ 0.03 ^{ab}	12.13 $\pm$ 0.19 ^{ab}	17.69 $\pm$ 0.71 ^a	372.50 $\pm$ 11.09 ^a	263.54 $\pm$ 10.01 ^{ab}	162.37 $\pm$ 5.24 ^a	22.47 $\pm$ 1.91 ^a	40.32 $\pm$ 0.94 ^{bc}
Preharvest salicylic acid	5.07 $\pm$ 0.13 ^{abc}	0.62 $\pm$ 0.01 ^a	12.08 $\pm$ 0.10 ^{ab}	19.41 $\pm$ 0.14 ^a	400.00 $\pm$ 27.39 ^a	257.09 $\pm$ 29.59 ^{ab}	177.55 $\pm$ 13.20 ^a	23.57 $\pm$ 1.87 ^a	34.04 $\pm$ 1.32 ^a
Preharvest oxalic acid	5.46 $\pm$ 0.13 ^c	0.60 $\pm$ 0.02 ^a	11.73 $\pm$ 0.32 ^{ab}	19.55 $\pm$ 1.01 ^a	387.50 $\pm$ 20.16 ^a	259.45 $\pm$ 14.18 ^{ab}	161.17 $\pm$ 8.36 ^a	25.31 $\pm$ 0.81 ^a	37.64 $\pm$ 1.12 ^{abc}
Postharvest salicylic acid	4.95 $\pm$ 0.09 ^{ab}	0.74 $\pm$ 0.03 ^b	12.23 $\pm$ 0.21 ^{ab}	16.61 $\pm$ 0.72 ^a	365.00 $\pm$ 17.08 ^a	269.65 $\pm$ 11.37 ^{ab}	166.95 $\pm$ 8.61 ^a	22.97 $\pm$ 1.22 ^a	41.60 $\pm$ 0.45 ^{bc}
Postharvest oxalic acid	4.97 $\pm$ 0.10 ^{ab}	0.70 $\pm$ 0.02 ^{ab}	12.50 $\pm$ 0.28 ^b	17.96 $\pm$ 0.74 ^a	382.50 $\pm$ 14.36 ^a	257.15 $\pm$ 9.49 ^{ab}	158.11 $\pm$ 3.27 ^a	23.36 $\pm$ 0.77 ^a	42.30 $\pm$ 1.07 ^c
Preharvest salicylic acid and postharvest salicylic acid	4.75 $\pm$ 0.09 ^a	0.64 $\pm$ 0.01 ^{ab}	12.28 $\pm$ 0.18 ^{ab}	19.17 $\pm$ 0.60 ^a	397.50 $\pm$ 25.29 ^a	213.52 $\pm$ 11.80 ^a	165.78 $\pm$ 6.55 ^a	27.56 $\pm$ 1.89 ^a	36.00 $\pm$ 0.89 ^{ab}
Preharvest salicylic acid and postharvest oxalic acid	5.35 $\pm$ 0.16 ^{bc}	0.63 $\pm$ 0.23 ^a	12.35 $\pm$ 0.25 ^{ab}	19.83 $\pm$ 0.81 ^a	425.00 $\pm$ 9.57 ^a	233.27 $\pm$ 28.42 ^a	164.58 $\pm$ 6.03 ^a	24.74 $\pm$ 1.39 ^a	37.77 $\pm$ 1.01 ^{abc}
Preharvest oxalic acid and postharvest salicylic acid	5.17 $\pm$ 0.11 ^{abc}	0.66 $\pm$ 0.03 ^{ab}	11.68 $\pm$ 0.11 ^{ab}	17.93 $\pm$ 0.82 ^a	372.50 $\pm$ 9.46 ^a	270.16 $\pm$ 9.78 ^{ab}	153.33 $\pm$ 4.08 ^a	25.66 $\pm$ 0.60 ^a	39.03 $\pm$ 1.14 ^{abc}
Preharvest oxalic acid and postharvest oxalic acid	5.09 $\pm$ 0.09 ^{abc}	0.65 $\pm$ 0.02 ^{ab}	11.38 $\pm$ 0.03 ^a	17.66 $\pm$ 0.62 ^a	375.00 $\pm$ 9.57 ^a	320.57 $\pm$ 23.71 ^b	163.56 $\pm$ 14.87 ^a	27.46 $\pm$ 1.50 ^a	37.11 $\pm$ 2.08 ^{abc}

Values are the means of four replicates $\pm$ standard error. Different letters in each column indicate significant differences between treatments at each assessment time (Tukey HSD, $p \leq 0.05$). FW = fresh weight.

Table 3. Weight loss, calyx stem-end rot, chilling injury score, overall acceptability score, colour, ethylene production and respiration rate of treated ‘Summerina’ mandarins assessed at harvest then stored for 10 weeks at 1 °C plus 1 week at 20 °C.

	Weight Loss (%)	Calyx Stem Rot (%)	Chilling Injury Score	Overall Acceptability Score	Colour (Chroma)	Colour (Hue)	Ethylene Production ($\mu\text{L C}_2\text{H}_4 \text{ kg}^{-1} \text{ h}^{-1}$)	Respiration Rate ($\text{mL CO}_2 \text{ kg}^{-1} \text{ h}^{-1}$)
Harvest								
Untreated				1.00 $\pm$ 0.00 ^a	66.31 $\pm$ 0.25 ^a	64.41 $\pm$ 0.24 ^b	ND	20.67 $\pm$ 0.44 ^a
Preharvest salicylic acid				1.00 $\pm$ 0.00 ^a	67.18 $\pm$ 0.21 ^a	65.25 $\pm$ 0.26 ^a	ND	22.55 $\pm$ 1.90 ^a
Preharvest oxalic acid				1.10 $\pm$ 0.00 ^a	66.92 $\pm$ 0.32 ^a	65.40 $\pm$ 0.22 ^a	ND	15.96 $\pm$ 3.38 ^a
10 weeks at 1 °C and 1 week at 20 °C								
Untreated	4.43 $\pm$ 0.46 ^{abc}	58.20 $\pm$ 2.60 ^c	1.58 $\pm$ 0.02 ^{ab}	2.85 $\pm$ 0.05 ^{abc}	62.88 $\pm$ 0.48 ^a	64.59 $\pm$ 0.20 ^a	0.78 $\pm$ 0.30 ^a	20.62 $\pm$ 1.05 ^a
Preharvest salicylic acid	3.60 $\pm$ 0.32 ^a	53.14 $\pm$ 5.32 ^{bc}	1.75 $\pm$ 0.02 ^{ab}	2.65 $\pm$ 0.06 ^a	64.13 $\pm$ 0.29 ^a	65.17 $\pm$ 0.21 ^a	0.70 $\pm$ 0.16 ^a	22.99 $\pm$ 2.30 ^a
Preharvest oxalic acid	3.77 $\pm$ 0.24 ^{ab}	47.39 $\pm$ 3.34 ^{abc}	1.75 $\pm$ 0.02 ^{ab}	2.83 $\pm$ 0.04 ^{abc}	64.00 $\pm$ 0.28 ^a	64.94 $\pm$ 0.19 ^a	1.41 $\pm$ 0.60 ^a	21.94 $\pm$ 0.96 ^a
Postharvest salicylic acid	4.69 $\pm$ 0.32 ^{bc}	28.39 $\pm$ 4.73 ^a	1.54 $\pm$ 0.02 ^a	2.88 $\pm$ 0.06 ^{abc}	63.44 $\pm$ 0.37 ^a	65.17 $\pm$ 0.26 ^a	0.52 $\pm$ 0.07 ^a	22.17 $\pm$ 0.69 ^a
Postharvest oxalic acid	4.54 $\pm$ 0.22 ^{abc}	35.51 $\pm$ 2.18 ^{ab}	1.55 $\pm$ 0.02 ^a	3.00 $\pm$ 0.06 ^{bc}	63.80 $\pm$ 0.32 ^a	64.78 $\pm$ 0.24 ^a	0.31 $\pm$ 0.18 ^a	21.94 $\pm$ 2.17 ^a
Preharvest salicylic acid and postharvest salicylic acid	3.93 $\pm$ 0.28 ^{abc}	37.25 $\pm$ 5.05 ^{ab}	1.87 $\pm$ 0.02 ^{ab}	2.90 $\pm$ 0.06 ^{abc}	64.27 $\pm$ 0.29 ^a	65.33 $\pm$ 0.18 ^a	0.56 $\pm$ 0.30 ^a	22.28 $\pm$ 1.61 ^a
Preharvest salicylic acid and postharvest oxalic acid	4.25 $\pm$ 0.28 ^{abc}	35.96 $\pm$ 2.39 ^{ab}	1.69 $\pm$ 0.02 ^{ab}	3.06 $\pm$ 0.05 ^{bc}	63.14 $\pm$ 0.48 ^a	64.70 $\pm$ 0.25 ^a	0.75 $\pm$ 0.22 ^a	18.97 $\pm$ 4.40 ^a

Table 3. Cont.

	Weight Loss (%)	Calyx Stem Rot (%)	Chilling Injury Score	Overall Acceptability Score	Colour (Chroma)	Colour (Hue)	Ethylene Production ($\mu\text{L C}_2\text{H}_4 \text{ kg}^{-1} \text{ h}^{-1}$)	Respiration Rate ($\text{mL CO}_2 \text{ kg}^{-1} \text{ h}^{-1}$)
Preharvest oxalic acid and postharvest salicylic acid	4.93 ± 0.37^c	42.94 ± 4.05^{abc}	1.50 ± 0.03^a	2.80 ± 0.07^{ab}	64.15 ± 0.27^a	65.42 ± 0.21^a	0.35 ± 0.13^a	20.97 ± 1.58^a
Preharvest oxalic acid and postharvest oxalic acid	4.36 ± 0.20^{abc}	51.39 ± 2.94^{bc}	1.96 ± 0.02^b	3.09 ± 0.07^c	63.09 ± 0.32^a	65.12 ± 0.21^a	0.14 ± 0.08^a	19.34 ± 0.21^a

Values are means of four replicates $\pm$ standard error. Different letters in each column indicate significant differences between treatments at each assessment time (Tukey HSD, $p \leq 0.05$). ND = none detected. Chilling injury score: 1 = normal (no pitting) and 4 = severe pitting (>30% surface coverage). Overall acceptability score: 1 = excellent condition and 5 = unacceptable, very soft.

Table 4. Firmness, titratable acidity (TA), total soluble solids (TSS), the maturity index (TSS:TA), vitamin C, ethanol content, peel antioxidant capacity (AOC), malondialdehyde content (MDA) and electrolyte leakage (EL) of treated ‘Summerina’ mandarins assessed at harvest then stored for 10 weeks at 1 °C plus 1 week at 20 °C.

	Firmness (kgf)	TA (% Citric Acid)	TSS (% Brix)	TSS:TA	Vitamin C ($\mu\text{L L}^{-1}$)	Ethanol ($\mu\text{L L}^{-1}$)	AOC ($\mu\text{mol TE g}^{-1} \text{ FW}$)	MDA ($\mu\text{mol kg}^{-1} \text{ FW}$)	EL (%)
Harvest									
Untreated	3.66 ± 0.05^a	0.89 ± 0.03^a	14.80 ± 0.08^a	16.77 ± 0.57^a	200.00 ± 9.13^a	ND	571.00 ± 15.68^a	27.50 ± 2.17^a	28.13 ± 0.47^a
Preharvest salicylic acid	3.74 ± 0.6^a	0.85 ± 0.03^a	14.60 ± 0.11^a	17.26 ± 0.03^a	197.50 ± 6.29^a	ND	526.02 ± 25.41^a	29.87 ± 3.29^a	27.52 ± 1.10^a
Preharvest oxalic acid	3.64 ± 0.04^a	0.80 ± 0.04^a	14.90 ± 0.2^a	18.83 ± 0.04^a	197.50 ± 2.50^a	ND	540.65 ± 16.63^a	30.16 ± 2.49^a	28.31 ± 0.59^a
10 weeks at 1 °C and 1 week at 20 °C									
Untreated	2.87 ± 0.04^{ab}	0.71 ± 0.02^a	14.80 ± 0.28^a	20.90 ± 0.79^a	87.50 ± 6.29^a	272.94 ± 16.15^a	179.93 ± 15.48^a	54.70 ± 1.26^a	39.58 ± 0.67^b
Preharvest salicylic acid	3.05 ± 0.06^{bcd}	0.74 ± 0.02^a	15.45 ± 0.27^a	20.83 ± 0.23^a	100.0 ± 9.13^a	258.99 ± 4.65^a	237.75 ± 16.29^a	53.17 ± 3.99^a	36.99 ± 0.84^{ab}
Preharvest oxalic acid	2.90 ± 0.04^{abc}	0.73 ± 0.04^a	15.45 ± 0.36^a	21.38 ± 0.76^a	82.50 ± 2.50^a	209.07 ± 16.57^a	185.22 ± 21.94^a	47.67 ± 2.31^a	37.06 ± 0.11^{ab}
Postharvest salicylic acid	2.88 ± 0.04^{abc}	0.80 ± 0.04^a	15.35 ± 0.44^a	19.23 ± 0.74^a	97.50 ± 4.79^a	263.29 ± 47.90^a	157.59 ± 12.52^a	50.77 ± 3.42^a	39.81 ± 0.47^b
Postharvest oxalic acid	2.86 ± 0.05^{ab}	0.78 ± 0.02^a	15.58 ± 0.18^a	20.12 ± 0.45^a	107.50 ± 10.31^a	213.60 ± 21.18^a	185.90 ± 16.99^a	46.42 ± 2.54^a	35.68 ± 0.48^a
Preharvest salicylic acid and postharvest salicylic acid	3.08 ± 0.06^{cd}	0.75 ± 0.02^a	15.13 ± 0.18^a	20.26 ± 0.42^a	92.50 ± 2.50^a	181.17 ± 33.39^a	220.35 ± 24.43^a	49.56 ± 2.37^a	39.48 ± 0.67^b
Preharvest salicylic acid and postharvest oxalic acid	3.13 ± 0.06^d	0.77 ± 0.02^a	15.73 ± 0.37^a	20.37 ± 0.62^a	100.0 ± 4.08^a	203.81 ± 10.04^a	224.45 ± 35.22^a	49.89 ± 3.01^a	38.40 ± 0.62^{ab}
Preharvest oxalic acid and postharvest salicylic acid	3.02 ± 0.05^{abcd}	0.75 ± 0.05^a	15.10 ± 0.22^a	20.44 ± 1.06^a	92.50 ± 6.29^a	325.97 ± 64.71^a	180.10 ± 23.57^a	49.23 ± 2.65^a	37.06 ± 0.82^{ab}
Preharvest oxalic acid and postharvest oxalic acid	2.83 ± 0.04^a	0.69 ± 0.01^a	15.08 ± 0.09^a	21.74 ± 0.34^a	105.0 ± 8.66^a	251.03 ± 57.95^a	213.36 ± 38.38^a	45.84 ± 2.82^a	38.17 ± 1.36^{ab}

Values are the means of four replicates $\pm$ standard error. Different letters in each column indicate significant differences between treatments at each assessment time (Tukey HSD, $p \leq 0.05$). ND = none detected. FW = fresh weight.

3.2.2. Physicochemical and Oxidative Parameters of ‘Summerina’ Mandarins

In ‘Summerina’ mandarins, no significant differences between treatments were observed for firmness, TSS, TA, maturity index (TSS:TA), vitamin C content, ethanol content, AOC, and MDA content at harvest or after 10 weeks at 1 °C followed by 1 week at 20 °C (Table 4). These findings are consistent with those observed in ‘Lane Late’ oranges, indicating that preharvest and postharvest applications of OA and SA did not substantially influence physicochemical or nutritional attributes under the storage conditions evaluated.

The peel AOC observation is contrary to the results for ‘Lane Late’ navel oranges (Table 2) and previous reports on lemons treated with OA (1 mM) and SA (0.5 mM) in the orchard [14,42,43]. The absence of consistent treatment effects suggests that responses to both SA and OA are cultivar- and species-dependent, and may also be influenced by application and timing.

In ‘Summerina’ mandarins, postharvest OA treatment resulted in lower EL (35.68%) compared with the untreated control (39.58%). However, no other significant differences were observed among the treatments. This reduction in EL may indicate improved membrane stability in response to postharvest OA applications [37]. However, this effect was not reflected in other oxidative stress indicators such as MDA or peel AOC.

Overall, these results indicate that preharvest and postharvest applications of OA and SA had limited effects on physicochemical and oxidative parameters in ‘Summerina’ mandarins under the storage conditions evaluated. These results contrast with the transient differences observed in peel AOC at harvest in ‘Lane Late’ oranges and previous reports in lemons treated with preharvest OA (1 mM) and SA (0.5 mM) [19,43], suggesting that responses to these treatments are cultivar- and species-dependent and influenced by application timing and storage conditions [14,30]. The observed reduction in EL following postharvest OA application indicates a potential improvement in membrane stability; however, this effect was not reflected in other oxidative stress indicators, suggesting that any treatment-induced responses were limited and not sustained. Overall, these findings support that while minor responses may occur, the application of OA and SA does not consistently affect internal fruit quality in ‘Summerina’ mandarins under prolonged cold storage conditions.

3.3. Comparative Postharvest Responses for ‘Lane Late’ Navel Oranges and ‘Summerina’ Mandarins

3.3.1. Minimal Responses Across Both Cultivars

The lack of consistent significant differences across most quality parameters suggests that both SA and OA had minimal impact on both cultivars’ fruit physiology under long storage condition in this study (1 °C for 10 weeks plus 1 week at 20 °C). Quality parameters discussed in Sections 3.1 and 3.2, including fruit colour, ethanol content, calyx condition and vitamin C content, were not significantly affected by either cultivar, suggesting minimal influence on visual quality, fermentative metabolisms and primary metabolic processes during extended cold storage [22,44,45]. This highlights the dominant influence of prolonged low-temperature storage in regulating fruit metabolic activity, which may have limited the expression of treatment-induced physiological responses [45,46].

3.3.2. Chilling Injury (CI) and Storage Stress

CI is a major storage disorder during long-term storage [8]. In this trial, CI was observed in both cultivars in Sections 3.1.1 and 3.2.1. SA and OA treatment did not affect CI for both cultivars; the symptoms were mild, with just slight pitting. Although prolonged storage at 1 °C can sometimes induce chilling stress in citrus fruit, the relatively low severity of CI observed suggests that the fruit itself or the level of stress imposed was insufficient

to elicit a strong physiological response to the applied treatments [12]. Consequently, the absence of significant treatment effects on CI should be interpreted with caution. Previous studies have reported reductions in CI following SA and OA application in citrus and other fruit systems [12–14,18,19]. However, such effects were not evident under the conditions used in this study. Additionally, treatment responses are known to vary with cultivar, maturity stage, and application conditions [10,12], which may explain the limited responses observed in both ‘Lane Late’ oranges and ‘Summerina’ mandarins. It is possible that treatment effects may become more apparent under severe chilling conditions or longer storage times.

3.3.3. Oxidative Responses

As observed in Sections 3.1.2 and 3.2.2, AOC and MDA content showed limited responses to SA and OA treatments in both cultivars following prolonged storage, indicating that oxidative status was not substantially altered. The oxidative response EL increased over the storage period in both cultivars. However, the effects of SA and OA treatments on EL varied between cultivars, where preharvest SA treatment was able to reduce EL in ‘Lane Late’ oranges and postharvest OA reduced EL in ‘Summerina’ mandarins. These responses suggest that treatment effects on membrane stability are dependent on both application timing and cultivar. However, they were not sufficient to induce consistent improvements in oxidative status or to influence CI development. As discussed in 3.1.2, previous studies have reported improved membrane stability and reduced EL following SA application [13,14]. However, such responses were not consistently observed in the present study. Furthermore, the lack of a consistent relationship between EL and visible CI symptoms highlights the complex nature of CI development, where changes in membrane integrity do not always correspond directly with external symptom expression [47].

3.3.4. Overall Implications

Overall, the results indicate that preharvest and postharvest applications of SA and OA had limited and inconsistent effects on fruit quality and physiological responses in both ‘Lane Late’ navel oranges and ‘Summerina’ mandarins during extended cold storage. While reductions in decay incidence were observed following postharvest treatments in mandarins, most quality parameters, including CI, AOC, membrane integrity, and internal quality attributes, were not significantly affected. These findings suggest that under prolonged low-temperature storage conditions, treatment-induced responses are limited and may depend on the level of physiological stress imposed [45]. No consistent synergised effects were observed when SA and OA were applied in combination. This was unexpected, as both compounds have been independently reported to enhance stress tolerance and reduce postharvest physiological disorders in horticultural crops [1,12,18,19]. The lack of synergistic responses may indicate that the physiological pathways influenced by SA and OA overlap, limiting any additive benefit when applied together. Alternatively, the concentrations or application timings used in the present study may not have been optimal to generate complementary responses. It is also possible that the relatively low level of chilling stress experienced by the fruit restricted the expression of treatment effects, thereby masking potential interactions between the two compounds. Further research investigating a wider range of concentrations, application schedules and storage conditions is recommended to determine whether combined SA and OA treatments can provide greater postharvest benefits. The variability in responses between cultivars and application methods further highlights the importance of fruit-specific characteristics and treatment timing in determining postharvest efficacy [10,12]. The present results indicate that their effectiveness under extended cold storage conditions may be limited and not economically

viable for industry. Therefore, additional strategies are required to improve long-term storage performance under commercial conditions, such as testing multiple concentrations, postharvest application, or combined interventions [46,48].

4. Conclusions

This study evaluated the effects of pre- and postharvest applications of SA and OA on the quality of ‘Lane Late’ navel oranges and ‘Summerina’ mandarins during extended cold storage. Overall, treatments had limited and inconsistent effects on most quality parameters, including CI, respiration rate, ethylene production, colour, vitamin C content, and AOC. Despite this, treatment-specific responses were observed. Postharvest SA treatment reduced stem-end rot in mandarins by 30%. Furthermore, EL was reduced with preharvest SA treatment in oranges and postharvest application of OA in mandarins. However, no consistent synergistic effects were observed between combined treatments. Treatment responses varied between citrus types, highlighting the importance of optimisation for each citrus type and cultivar. The relatively low incidence of CI observed during storage may have contributed to the limited treatment responses detected, suggesting that the storage conditions imposed insufficient chilling stress to fully determine the effects of SA and OA treatments against CI. Therefore, the absence of significant treatment effects on CI should be interpreted with caution. Overall, the results indicate that SA and OA provide limited but targeted benefits during extended cold storage. New strategies and future research should focus on testing multiple OA and SA concentrations, earlier preharvest application, and integration with other postharvest strategies, such as hot water dipping, to improve citrus fruit quality under long cold storage conditions.

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Conflicts of Interest: The authors declare that there are no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

SA	Salicylic acid
OA	Oxalic acid
CI	Chilling Injury
TSS	Total soluble solids

TA	Titrateable acidity
DPPH	2,2-diphenyl-1-picrylhydrazyl
MDA	Malondialdehyde
EL	Electrolyte leakage
AOC	Antioxidant capacity
ROS	Reactive oxygen species
CAT	Catalase
POD	Peroxidase
APX	Ascorbate peroxidase

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