Effect of Pre-Harvest Oxalic Acid Treatment on Shelf-life of Apricot cv. ‘Roxana’

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Abstract
The effect of pre-harvest oxalic acid treatment on the shelf-life of apricot cv. ‘Roxana’ was investigated. For this purpose, different doses (0, 1, 2, 4, and 8 mM) of oxalic acid (OA) solutions were applied 7 days before the optimal harvest stage. Fruit, harvested at optimum stage (firm-ripe stage), transported to the postharvest physiology laboratory, immediately. Harvested fruit was kept at room (20±1°C) temperature and 50–60% relative humidity conditions for 8 days for shelf-life evaluation. During the storage period, some physical and chemical analyses (weight loss, fruit firmness, soluble solids content, titratable acidity, fruit color, ethylene production, and respiration rate) were performed at 2-day intervals. As a result, all doses of OA gave better results than the control group in terms of some quality parameters. Especially, 1 mM dose of OA was the most effective treatment for maintaining fruit quality.

Keywords: Oxalic acid; apricot; shelf-life; quality

Hasat Öncesi Oksalik Asit Uygulamasının ‘Roxana’ Kayısı Çeşidinin Raf Ömrü Üzerine Etkisi

Öz
Çalışmada, hasat öncesinde oksalik asit (OA) uygulamasının raf ömrü süresince ‘Roxana’ kayısı çeşidinin meyve kalitesi üzerine etkisi araştırılmıştır. Bu amaçla, tahmini hasat tarihinden 7 gün önce farklı dozlarda (0, 1, 2, 4 ve 8 mM) oksalik asit uygulanmıştır. Optimum hasat tarihinde (sert olum döneminde) derilen meyveler oda sıcaklığında (20±1°C) ve %50–60 nem koşullarında 8 gün süreyle raf ömründe tutulmuştur. Raf ömrü süresince 2 gün aralıklarla bazı fiziksel ve kimiyasal analizler (ağırlık kaybı, metve eti sertliği, suda çözünerebilir kuru madde miktarı, titre edilebilir asitlik, meyve rengi, etilen üretimi ve solunum hızı) yapılmıştır. Sonuç olarak, farklı dozlardaki OA uygulamalarının hepsi bazı meyve kalite parametrelerinde kontrolden daha iyi sonuçlar vermiştir. Özellikle OA’nın 1 mM’lik dozu meyve kalitesinin korunması bakımından en iyi uygulama olduğu saptanmıştır.

Anahtar Kelimeler: Oksalik asit; kayısı; raf ömrü; kalite

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1. Introduction

Apricot is considered one of the most popular and common fruit in the world [1]. Apricot fruit has sufficient amounts of sucrose, glucose, fructose, antioxidant components (lycopene, β-carotene, vitamins A, and E) and minerals (K, P, Mg). Consumption of apricots plays an important role in preventing diseases and maintaining a healthy life [2]. Apricots, as climacteric stone fruit, have a limited postharvest life. The main factor limiting the postharvest life of apricot is related to ripening characteristics of the fruit. Apricot is a highly perishable climacteric fruit, which suffers rapid ripening and deterioration after postharvest, and thus has a limited postharvest life at room temperature [3, 4].

During postharvest life, due to internal and external factors, chemical and physical changes occur in fruit and vegetables, which result in losses in nutritional and sensory quality [5]. One of the recommended methods for preserving fruit quality after harvest is the application of oxalic acid (OA). Oxalic acid is an organic acid distributing widely in various organisms, especially in plants [6]. In previous studies, it has been shown that the postharvest application of exogenous OA has received much attention, mainly due to delay fruit ripening, acts as anti-senescence and anti-browning agent with high antioxidant properties [7]. The postharvest life extension of mango and peaches fruit by oxalic acid also has been reported when 1 to 5 mmol/L of concentrations were applied [6]. Also, both pre- and postharvest OA treatments for extending shelf-life and maintaining postharvest quality of fruit and vegetables have been investigated in different fruit species. It was reported that the non-toxic concentrations of OA reduced enzymatic browning [8], controlled diseases and decays [6 and 9], prolonged shelf life [10], delayed chilling injury [11 and 12], and slowed down respiration rate and ethylene production [13 and 14] of some horticultural crops. Besides, it has been stated that the anti-senescence and stress-protective effect of OA can be associated with its antioxidant characteristic and regulation of ethylene signals [6]. The OA contributed to the delay of ripening and loss of fruit firmness by inhibiting ethylene production and respiration rate in mango, plum, jujube, peach, pear, apple, and banana [13, 10, 15, 12, 6, 16 and 7]. However, as far as we know, no detailed investigations of the effect of pre-harvest OA treatment on the shelf life of Roxana apricot at room temperature were carried out.

The objective of this study was to investigate the effect of pre-harvest oxalic acid treatment on the shelf-life of apricot cv. ‘Roxana’ at room temperature (20±1°C).

2. Material and Method

Experiments were performed on 8-years old ‘Roxana’ apricot trees. Apricot trees planted in the north-south row direction; between row distances 7×7 m, were located in Isparta-Turkey. All the cultural practices including pruning, irrigation, pesticide spray, etc. had been assessed for several years as periodically. OA was applied as an aqueous solution, containing 1 % non-ionic surfactant (Tween-20), onto fruit and leaves around until runoff. For this purpose, different doses (0, 1, 2, 4, and 8 mM) of OA solution were applied 7 days before optimal harvest time. Control trees only received an aqueous solution containing the same concentration of surfactant. Apricot fruit at a uniform size, free from visual symptoms of disease or blemishes, were harvested at commercial maturity. Fruit, harvested at optimum stage (firm-ripe stage), transported to the postharvest physiology laboratory, immediately. Harvested fruit was kept at 20±1°C °C temperature and 50–60% relative humidity conditions for 8 days for shelf-life evaluation. Weight loss (%), fruit flesh firmness (N), soluble solids content (%), titratable acidity (%), fruit skin color (CIE L*a*b*), ethylene production (µl/kg.h) and respiration rate (ml CO₂/kg.h), were determined at the beginning of the storage and 2- day intervals during the storage period. Data were subjected to analysis of variance (ANOVA, JMP7), means were separated by Tukey test (P<0.05).
2.1. Chemical and physical analysis

Weight loss: Weight loss of apricots was measured over 15 fruit in each replicate and expressed as the percentage of loss of weight concerning the initial weight. Weight loss was determined by the formula; [(First weight - Last weight) / First weight] × 100

Fruit flesh firmness: Firmness was measured over 15 fruit in each replicate. Fruit firmness was determined using a digital texture machine (Lloyd Instruments LF Plus) and measured via compression using a 50 N load cell and a stainless steel, 5.1 mm diameter cylindrical probe with a constant speed of 100 mm min⁻¹ at harvest date and during storage periods. The maximum force generated during the probe travel was used for data analysis. The results were expressed as Newton (N).

Respiration rate and ethylene production: Respiration rate and ethylene production were measured in 500 grams of fruit samples for each replicate. The fruit was weighed and placed in 2 L airtight jars at 20 °C for 1-2 hours. Then the gas sample was taken from jars and injected into gas chromatographs. Measurements were made in split/splitless (S/SL) of an inlet in split mode with gas sampling valve with 1 ml gas sample by using fused silica capillary column (GS-GASPRO, 30 m × 0.32 mm I.D., U.S.A), with thermal conductivity detector (TCD) for respiration rate measurements and flame ionization detector (FID) for ethylene production measurements by Agilent GC-6890N (U.S.A and Canada) model gas chromatography (GC) and Chemstation A.09.03 [1417] software. Carrier gas flow was 1.7 ml/min in stable flow mode. The temperature of the oven, TCD, and FID detectors were 40 °C (isothermal), 250 °C and 250 °C, respectively. Results were expressed as μl/kg.h for ethylene production and ml CO₂/mL/kg.h for respiration rate.

Soluble solids content and titratable acidity: Soluble solids content (SSC) was measured using a digital refractometer (Atago Pocket PAL-1) and expressed as a percentage (%). Titratable acidity (TA) was determined by a digital pH meter (Hanna Instruments HI 9231) and trimeter (Digital, Isolab), and expressed as a percentage (%).

Fruit skin color: Fruit skin color was determined using a colorimeter (Minolta Cr 300, Ramsey, NJ, USA) over 15 fruits in each replicate. Minolta color measurement apparatus was calibrated according to the standard white calibration plate (Y = 92.3, x = 0.3136 and y = 0.3194). The values were expressed by the CIE L* (brightness-darkness), a* (+ a*: red, − a*: green), and b* (+ b*: yellow, − b*: blue) system.

3. Results

3.1. Weight loss

As shown in Table 1, fruit weight loss was significantly increased in apricot from day 2 to day 8 of shelf life at 20 °C irrespective of the treatments. The highest average weight loss (3.69 %) value was obtained from the control fruit during storage. At the end of the shelf-life period (at day 8), the lowest fruit weight loss (5.31%) was observed in fruit treated with 8 mM OA compared to other treatments. Among all treatments, the lowest average fruit weight loss (3.11 %) was determined in 8 mM OA-treated fruit, while the highest one (3.69 %) was obtained from control fruit.
Table 1. Weight loss (%) of ‘Roxana’ apricots treated with OA during shelf life.

| Treatments | Shelf life (days) | 2 | 4 | 6 | 8 | Means |
|------------|------------------|---|---|---|---|-------|
| Control    | 0.95±0.06        | 3.11±0.05 | 4.65±0.50 | 6.03±0.32 | 3.69 NS |
| 1 mM       | 0.87±0.08        | 2.47±0.16 | 4.01±0.38 | 5.79±0.44 | 3.29 |
| 2 mM       | 0.88±0.01        | 2.58±0.08 | 4.05±0.44 | 5.47±0.02 | 3.25 |
| 4 mM       | 0.84±0.04        | 2.83±0.05 | 4.10±0.24 | 5.61±0.04 | 3.35 |
| 8 mM       | 0.88±0.06        | 2.38±0.42 | 3.86±0.19 | 5.31±0.18 | 3.11 |
| Means      | 0.89 d           | 2.67 c   | 4.13 b   | 5.64 a    |       |

*: Means followed by the same letter in the same column are not statistically significant (P<0.05). NS: non-significant. Values are mean ± standard deviations (n=3)

3.2. Fruit flesh firmness

All concentrations of OA affected fruit flesh firmness. The flesh firmness of all fruit gradually decreased with increasing storage time, but higher flesh firmness values were observed in OA treated fruit compared to control during shelf life. The highest fruit flesh firmness (19.23 N) was detected in 1 mM OA-treated fruit, whereas untreated fruit gave the lowest (16.13 N) one (Table 2).

Table 2. Fruit flesh firmness (N) of ‘Roxana’ apricot treated with OA during shelf life.

| Treatments | Shelf life (days) | 0 | 2 | 4 | 6 | 8 | Means |
|------------|------------------|---|---|---|---|---|-------|
| Control    | 51.05±4.60       | 19.03±10.26 | 6.12±3.70 | 3.07±0.78 | 2.36±0.93 | 16.13 b* |
| 1 mM       | 52.63±4.34       | 29.31±8.88 | 6.00±0.94 | 4.35±0.97 | 3.85±0.47 | 19.23 a |
| 2 mM       | 54.56±6.79       | 20.55±8.70 | 5.44±1.42 | 3.70±0.85 | 3.13±0.46 | 17.48 ab |
| 4 mM       | 52.47±5.10       | 18.43±6.49 | 5.48±1.03 | 4.35±0.71 | 2.47±0.66 | 16.72 b |
| 8 mM       | 54.10±7.84       | 19.92±3.26 | 4.65±1.20 | 4.32±0.98 | 3.57±0.64 | 17.31 ab |
| Means      | 52.96 a*         | 21.45 b   | 5.54 c    | 3.96 cd   | 3.15 d    |       |

*: Means followed by the same letter in the same row and column are not statistically significant (P<0.05). Values are mean ± standard deviations (n=15)

3.3. Soluble solids content (SSC) and titratable acidity (TA)

The effects of OA treatments on SSC and TA were statistically significant (P<0.05). The SSC fluctuated and increased at the end of 8 days of storage compared to initial values in all treatments. The highest SSC value (12.45 %) was determined from untreated fruit, whereas the lowest one (10.79 %) was detected in 1 mM OA-treated fruit. As expected, the increase in SSC of fruit during the storage is thought to be due to the use of water loss and the ripening process. It is known that the SSC of climacteric fruit increases, as percent, with storage time depending on water loss. The TA contents of fruit gradually decreased over the storage period regardless of treatments. The highest TA value was obtained from a 1 mM dose of OA throughout shelf life (Table 3).

Table 3. SSC (%) and TA (%) of ‘Roxana’ apricots treated with OA during shelf life.

| Treatments | Shelf life (days) | SSC (%) | Means |
|------------|------------------|---------|-------|
| Control    | 0 | 2 | 4 | 6 | 8 | Means |
| Control    | 12.13±0.45 | 11.83±0.31 | 12.97±0.31 | 12.37±0.19 | 12.97±0.68 | 12.45 a* |
| 1 mM       | 9.87±0.05 | 10.60±0.08 | 11.53±0.42 | 11.27±0.37 | 10.70±0.50 | 10.79 d |
| 2 mM       | 10.27±0.09 | 11.43±0.12 | 12.40±0.16 | 11.17±0.12 | 11.10±0.22 | 11.27 c |
| 4 mM       | 11.57±0.05 | 11.33±0.29 | 12.37±0.33 | 12.40±0.08 | 11.67±0.12 | 11.82 b |
| 8 mM       | 12.17±0.33 | 11.00±0.14 | 12.30±0.16 | 11.27±0.12 | 12.63±0.05 | 11.87 b |
| Means      | 11.20 c*   | 11.24 c   | 12.31 a   | 11.69 b    | 11.81 b    |       |

| Treatments | TA (%) |
|------------|--------|
| Control    | 1.92±0.02 | 1.56±0.21 | 1.35±0.12 | 1.36±0.07 | 1.22±0.26 | 1.48 a |

*: Means followed by the same letter in the same column are not statistically significant (P<0.05). Values are mean ± standard deviations (n=3)
3.4. Ethylene production and respiration rate

In the present study, the effect of the treatments on ethylene production (μl/kg.h) and respiration rate (ml CO$_2$/kg.h) was significant (P<0.05). The highest ethylene production (66.97μl/kg h) was determined from untreated fruit, whereas fruit treated with 4 mM OA gave the lowest ethylene production (48.59 μl/kg.h). The impact of OA on the development of ethylene production, in general, was concentration-dependent. In other words, the average ethylene production of fruit decreased with increasing doses of OA except for 8 mM showing its obvious effect on ethylene biosynthesis (Table 4).

### Table 4. Ethylene production (μl/kg.h) and respiration rate (ml CO$_2$/kg.h) of ‘Roxana’ apricots treated with OA during shelf life.

| Treatments | Shelf life (days) | 0       | 2       | 4       | 6       | 8       | Means  |
|------------|-------------------|---------|---------|---------|---------|---------|--------|
| Ethylene production |                   |         |         |         |         |         |        |
| Control    |                   | 1.27±0.42 | 5.61±0.41 | 80.69±2.86 | 225.06±19.64 | 22.21±2.83 | 66.97 a* |
| 1 mM       |                   | 0.79±0.09 | 3.88±0.24 | 75.25±4.73 | 161.66±9.06 | 51.98±4.02 | 58.71 ab |
| 2 mM       |                   | 0.63±0.06 | 3.49±0.80 | 73.56±4.21 | 156.35±0.62 | 56.27±0.22 | 58.06 ab |
| 4 mM       |                   | 0.91±0.02 | 2.72±0.13 | 39.14±3.34 | 138.54±7.42 | 61.65±3.90 | 48.59 b |
| 8 mM       |                   | 0.74±0.11 | 3.79±0.32 | 69.98±10.09 | 143.67±1.56 | 34.07±2.93 | 50.45 b |
| Means      |                   | 0.87 d*  | 3.90 d   | 67.72 b  | 165.06 a  | 45.24 c  |        |
| Respiration rate |                 |         |         |         |         |         |        |
| Control    |                   | 40.70±3.69 | 32.96±1.39 | 65.97±0.67 | 283.94±12.59 | 31.37±3.41 | 90.99 a |
| 1 mM       |                   | 43.85±2.60 | 35.48±3.44 | 64.57±1.03 | 156.29±17.41 | 29.70±5.44 | 65.49 b |
| 2 mM       |                   | 37.53±3.56 | 56.68±3.87 | 65.55±5.70 | 167.24±9.40 | 30.27±4.25 | 71.46 b |
| 4 mM       |                   | 43.35±6.55 | 53.66±1.55 | 63.13±8.92 | 137.27±17.81 | 30.06±5.07 | 59.22 b |
| 8 mM       |                   | 35.29±2.69 | 47.69±5.36 | 62.91±8.89 | 126.26±17.41 | 23.93±6.24 | 65.98 b |
| Means      |                   | 40.70 cd* | 40.14 c  | 45.29 b  | 64.43 a   | 174.20 d  | 29.07  |

*: Means followed by the same letter in the same row and column are not statistically significant (P<0.05). Values are mean ± standard deviations (n=3)

3.5. Fruit skin color

The skin color results of both sides (red and yellow) of fruit are presented in Tables 5 and 6. The effects of OA treatments and storage periods on L*, a*, b* values of skin color were statistically (P<0.05) significant. The highest L* and b* values were obtained from 1 mM OA-treated fruit as 62.21, 44.25, respectively. All treatments showed similar characteristics of the a* value when compared to the control group. The lowest a* (17.27) value was obtained from 1 mM OA treated fruit (Table 5).

### Table 5. Change color (L*,a*,b*) of ‘Roxana’ apricots treated with OA during shelf life (red side of fruit).

| Treatments | Shelf life (days) | 0       | 2       | 4       | 6       | 8       | Means  |
|------------|-------------------|---------|---------|---------|---------|---------|--------|
| Control    |                   | 47.85±2.98 | 55.85±4.49 | 54.07±6.46 | 52.07±6.46 | 50.07±6.46 | 51.98 bc* |
| 1 mM       |                   | 61.31±4.18 | 65.07±3.94 | 63.56±4.15 | 61.56±4.15 | 59.56±4.15 | 62.21 a |
| 2 mM       |                   | 50.84±2.07 | 54.30±3.54 | 53.73±3.65 | 51.73±3.65 | 49.73±3.65 | 52.06 bc |
| 4 mM       |                   | 50.16±2.66 | 52.83±3.69 | 50.86±3.57 | 48.86±3.57 | 46.86±3.57 | 49.91 c |
| 8 mM       |                   | 51.25±2.14 | 55.83±4.47 | 53.90±2.55 | 51.90±2.55 | 49.90±2.55 | 52.56 b |
| Means      |                   | 52.28d*  | 56.78a   | 55.22 b  | 53.22 c  | 51.22 d  |        |

*: Means followed by the same letter in the same row and column are not statistically significant (P<0.05). Values are mean ± standard deviations (n=3)
L* value, which shows fruit brightness of fruit skin, decreased during the storage period. The a* and b* value of the fruit generally showed an increase during shelf life, while L* value decreased throughout 8 days except for the control sample. The highest L* and b* values were obtained from 1 mM OA-treated fruit as 67.76 and 59.85, respectively. The lowest a* (3.02) value was obtained from 1 mM OA treated fruits as found on the red side of fruit (Table 6).

4. Discussion

As shown in Table 1, fruit weight loss was significantly increased in apricot from day 2 to day 8 of shelf life at 20°C irrespective of the treatments. Among all treatments, the lowest average fruit weight loss (3.11%) was determined in 8 mM OA-treated fruit, while the highest one (3.69%) was obtained from control fruit. Fruit lost their weight mainly due to respiration and transpiration through the skin and various metabolic activities. OA treatments decreased the weight loss in mango fruit, as compared to control during cold storage [7]. In accordance with our results, previous researchers reported that pre and postharvest OA treatments decreased weight loss of fruit in parallel with increasing storage time [17,18]. Also, both pre- and postharvest OA treatments. Flesh firmness of all fruit gradually decreased with increasing...
storage time, but higher flesh firmness values were observed in OA treated fruit compared to control during shelf life. Fruit softening rate was slowed down by OA during storage at room temperature [10]. There was a continuous decline in fruit flesh firmness of peaches during the storage period, but the amount of decrease in OA-treated fruit was less than non-OA-applied fruit [5]. They suggested that the inhibition of softening was associated with decreased polygalacturonase (PG) and pectin methylesterase (PME) activities; that is, the retardation of pectin solubilization/degradation. Flesh firmness of plum was maintained, and the shelf life of fruit was extended significantly by postharvest oxalic acid application [6]. The SCC consists largely of sugars and, the changes in SCC during storage are caused by changes in carbohydrate structure [22]. The SCC can vary according to the maturity stages of the fruit, and it is known that the SCC in ripe fruit is higher than that of unripe ones. The SCC fluctuated and increased at the end of 8 days of storage compared to initial values in all treatments. SSC and SSC/TA ratio were significantly increased, while TA and ascorbic acid contents decreased regardless of the treatments in mango fruit [7]. In this study, it was also indicated that the increase or decrease in biochemical attributes of mango was significantly delayed by OA treatments. SSC in the control group increased to a maximum on a ninth day, then declined slightly for the rest of the storage period, but SSC in pre-OA treated kiwifruit increased gradually during storage at room condition [19]. In the present study, the effect of the treatments on ethylene production (μl/kg.h) and respiration rate (ml.CO₂/kg.h) was significant (P<0.05). The impact of OA on the development of ethylene production, in general, was concentration-dependent. In other words, the average ethylene production of fruit decreased with increasing doses of OA except for 8 mM showing its obvious effect on ethylene biosynthesis (Table 4). Similarly, the ethylene synthesis of OA treated fruit was higher than those of untreated ones during storage period time [6, 9]. Respiration rate is an important factor for maintaining fruit quality during cold storage and shelf life. OA treatment significantly suppressed and reduced endogenous ethylene production [6]. The reduced ethylene production in OA-treated fruit might be ascribed to the reduced 1-aminocyclopropane-1-carboxylic acid synthase (ACS) activity [7]. Also, it was reported that the exogenous application of OA suppressed the ethylene production and respiration rate in banana, plum, and mango fruits [20, 7]. The skin color values varied depending on ripening but were delayed, relatively, by OA treatments on both sides of the fruit. Similarly, the skin color values of fruit were affected by OA applications [21].

5. Conclusions

In conclusion, all doses of OA gave better results than the control group in terms of some quality parameters. Especially, 1 mM dose of OA was the most effective treatment for maintaining fruit quality. The results suggest that OA has the potential to extend the shelf life of apricot by delaying quality loss.

6. References

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