Quality of ‘Valencia Delta’ orange after degreening and coating with wax

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A B S T R A C T
In this study, oranges of the cultivar 'Valencia Delta' were degreened, coated with carnaúba-based wax and stored under refrigeration. The influence of the application of exogenous ethylene and coating was observed on physical and physico-chemical properties of the fruits. After the application of the treatments, the fruits were refrigerated (7 ± 2 °C) for a period of 35 days. The analyzed variables included weight loss, peel color, titratable acidity, ascorbic acid, total soluble sugars, reducing sugars, total polyphenols and total chlorophyll of the peel. The coated fruits showed significant reduction in weight loss when compared with the uncoated fruits, which lost about 3.0% of moisture at the end of the experiment. Significant reduction in the values of hue angle, accompanied by sharp deterioration in chlorophyll contents, suggested that the applied ethylene concentration was efficient in reducing green peel color. The contents of total polyphenols, total soluble sugars and reducing sugars increased while ascorbic acid decreased. The application of the coating, after degreening, was fundamental in maintaining the quality of the 'Valencia Delta' orange during storage.

Key words: postharvest exogenous ethylene coating storage

Qualidade de laranja ‘Valência Delta’ após desverdecimento e recobrimento com cera

R E S U M O
Laranjas da cultivar ‘Valência Delta’ foram, no presente trabalho, desverdecidas, recobertas com cera à base de carnaúba e armazenadas sob refrigeração. Observou-se a influência da aplicação do etileno exógeno e do recobrimento nas propriedades físicas e físico-químicas dos frutos; após a aplicação dos tratamentos os frutos foram refrigerados (7 ± 2 ºC) durante 35 dias; as variáveis analisadas incluíram perda de massa, coloração da casca, acidez titulável, ácido ascórbico, açúcares solúveis totais e redutores, polifenóis totais e clorofila total da casca. Os frutos recobertos apresentaram redução significativa na perda de massa quando comparados com os frutos não recobertos que perderam aproximadamente 3,0% de umidade ao final do experimento. Redução significativa nos valores de ângulo hue acompanhada de degradação acentuada nos teores de clorofila sugeriu que a concentração de etileno aplicada foi eficiente na redução da coloração verde da casca. Os teores de polifenóis totais, açúcares solúveis totais e redutores aumentaram enquanto o de ácido ascórbico decresceu. A aplicação de recobrimento após o desverdecimento foi fundamental na manutenção da qualidade da laranja ‘Valência Delta’ durante o armazenamento.
Introduction

The Brazilian agro-industry stands out in the production of citrus, especially orange, and the Northeast region is the second largest producer. ‘Valência’ orange is sweet, typical of subtropical countries and economically important due to its high yield and quality, for industrial processing and fresh consumption (Oliveira et al., 2008). Citric fruits are sources of vitamins, fibers and antioxidant compounds, such as ascorbic acid, phenolic compounds and flavonoids (Jayaprakasha & Patil, 2007).

Among the quality variables for the fresh consumption of fruits, fruit size, peel color intensity, firmness, appearance, content of sugars and proportion of total acids stand out. Period and site of harvest, variety, cultivation practices and post-harvest handling influence these characteristics (Rodolfo Júnior et al., 2007).

Post-harvest techniques, such as refrigeration, utilization of coating and application of ethylene, have been used to avoid physical and physicochemical modifications and losses of quality during storage. Refrigeration is the technology most used in the conservation of fruits and vegetables, because it reduces metabolic activities, production of ethylene, alterations in the composition, senescence speed and loss of water, prolonging the shelf life.

The application of coating has been used to preserve the quality and extend the shelf life of the fruits, reducing water loss and promoting brightness (Cantillano et al., 2009). Additionally, to cater to the consumer, techniques to modify the external appearance of the fruit can be used, such as the application of ethylene during storage. Exogenous ethylene alters peel color, since it accelerates chlorophyll degradation, promotes the appearance of carotenoids and increases their synthesis (Rodrigo & Zacarias, 2007).

Therefore, it becomes necessary to analyze physical and physicochemical parameters of orange, cv. ‘Valência Delta’, with different post-harvest techniques. Thus, this study aimed to evaluate the quality of ‘Valência Delta’ orange subjected to degreening followed by coating and cold storage.

Material and Methods

Fruits of ‘Valência Delta’ orange [Citrus sinensis (L.) Osbeck], ‘Swingle’ citrulmo rootstock [Citrus paradisi Macfad. x Poncirus trifoliata (L.) Raf.], were harvested in the Baixo Jaguaribe region, in the municipality of Limoeiro do Norte, CE, Brazil. The fruits showed mean weight of 323 g, diameter of 80.04 mm and hue angle of 125.00, and were harvested in the morning, placed in cardboard boxes and transported to the Federal University of Ceará, Department of Food Technology. The fruits were selected regarding the maturation stage, variety, cultivation practices and post-harvest handling influence these characteristics (Rodolfo Júnior et al., 2007).

Physicochemical parameters of orange, cv. ‘Valência Delta’, were expressed in percentage. The content of ascorbic acid was determined according to the methodology described by Yemm & Willis (1954); readings were obtained in spectrophotometer at 620 nm and the results were expressed in (mg ascorbic acid 100 g of juice). The total soluble sugars were determined according to the methodology described by Yemm & Willis (1954); readings were obtained in spectrophotometer at 620 nm and the results were expressed in percentage. Reducing sugars were determined according to the methodology described by Miller (1959); readings were performed in spectrophotometer at 540 nm and the results were expressed in percentage.

The content of total polyphenols was determined through titration with 0.1 M NaOH solution (IAL, 2008) and the results were expressed in percentage of citric acid. The content of ascorbic acid was determined according to the methodology described by Strohecker & Henning (1967) and the results were expressed in (mg ascorbic acid 100 g of juice). The total soluble sugars were determined according to the methodology described by Yemm & Willis (1954); readings were obtained in spectrophotometer at 620 nm and the results were expressed in percentage. Reducing sugars were determined according to the methodology described by Miller (1959); readings were performed in spectrophotometer at 540 nm and the results were expressed in percentage.
The experimental design was completely randomized in split plot scheme. The main plot was composed of the treatments (fruits exposed to ethylene and control, with and without coating) and the subplots corresponded to storage periods (0, 7, 14, 21, 28 and 35 days). The results were subjected to analysis of variance (F test) and the determination of minimum significant difference was evaluated by Tukey test (p < 0.05). Regression analyses used polynomials until the 3rd degree and the minimum value of $R^2$ was 70%.

**Results and Discussion**

The use of coating in ‘Valência Delta’ orange showed relevant effect, since it reduced the weight loss and promoted better visual quality in the fruits during storage. According to Figure 1, the weight loss of ‘Valência Delta’ orange increased during the storage in all treatments; however, there was no significant difference (p < 0.05) between the treatments, except the control, which showed higher weight loss, a value higher than 3.0%, at the end of the storage period.

The fruits coated with wax, regardless of the ethylene application, showed lower weight loss. Thus, it could be observed that the application of coating in the maintenance of fruit weight showed satisfactory effect and that the application of exogenous ethylene did not interfere significantly with the weight loss of the fruits.

Chaudhary et al. (2012) evaluated degreened ‘Star Ruby’ pomelos and observed minimum weight loss (3.26%) after 35 days of storage.

In the evaluation of peel color, the hue angle (Figure 2) initially showed a value of 125, decreasing during the storage period. The treatment with exogenous ethylene in the fruits was significant (p < 0.05), because the accentuated decrease in hue values became noticeable, associated with the increase in chroma values.

The use of coating retarded the development of the yellow color on the peel of fruits that were not exposed to ethylene. Even in fruits exposed to ethylene and coated with wax, this characteristic was observed, because, despite the change in peel color, these fruits showed higher hue values due to the use of coating. Similar results were reported by Machado et al. (2015), who observed decrease in the values of hue angle.

Degreening treatment (D) and coating (R) were highly effective in maintaining the peel color of the fruits, since the green color of flavedo was preserved. Fruits exposed to ethylene showed higher chroma values, indicating higher color intensity; in this case, yellow. These values were related to the decrease in hue angle and to the increment in chroma, confirming the change from the green color to yellow. In oranges that were not exposed to ethylene, the chroma value remained virtually unchanged during the storage period; thus, it can be claimed that the green color of the flavedo was preserved.

According to Figure 4, luminosity was influenced by the treatments and storage periods, exhibiting significant difference (p < 0.05). The action of the exogenous ethylene could be observed on the process of degreening of the fruits, because the values of luminosity were initially close to 40 and, after degreening, they increased to 53, indicating that the color of fruit peels became lighter or brighter.

Thus, fruits exposed to ethylene showed higher values of luminosity, indicating lighter peel color. Fruits that were not degreened (RD) and coated (R) showed higher luminosity values, which were maintained throughout the storage period. The minimum weight loss was achieved in these treatments, which can be related to the better visual quality in the fruits during storage. According to Figure 3, the coating treatment (R), regardless of the degreening (D), showed lower weight loss. Thus, it could be claimed that the green color of the flavedo was preserved.

The chromaticity of the flavedo, which indicates the intensity of the color, was also influenced by the application of coating (Figure 3). There was significant difference (p < 0.05) between the treatments and the storage period.

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exposed to ethylene also showed increase in luminosity, but in a less intense way. Mayuoni et al. (2011), analyzing citric fruits, also observed that the application of exogenous ethylene accelerated the color change in fruit peels.

Total chlorophyll decreased during the storage period in all applied treatments (Figure 5). Due to the degreening process, the fruits that were exposed to exogenous ethylene exhibited greater reduction in the values of total chlorophyll, because ethylene accelerated its degradation process. Covered fruits showed lower chlorophyll degradation due to the use of coating, which contributed to a lower degradation.

The application of degreening in citric fruits induced the degradation of green pigments (chlorophyll) and increased the synthesis of carotenoids, changing the color of the peel (Blum & Ayub, 2008). Rodrigo & Zacarias (2007) also observed reductions in the contents of chlorophyll of the peel of ‘Navelate’ orange degreened with ethylene.

In the evaluation of titratable acidity (Table 1), there were statistical differences (p < 0.05) between the treatments after 28 days of storage. ‘Valencia Delta’ orange showed acidities around 0.6%. It was observed that the values remained statistically equal along the storage period in all treatments. Rapisarda et al. (2008) observed that, under different climatic conditions, there was increase in the contents of citric acid in the variety ‘Valéncia’, in study conducted with five different genotypes of orange.

Mayuoni et al. (2011) used exogenous ethylene for the degreening of citric fruits at 20°C and observed that there was no influence on the acidity content. Tietel et al. (2010) claimed that the utilization of exogenous ethylene for degreening at moderate temperatures of approximately 20°C did not damage the taste of the fruit, while in citric fruits the degreening at high temperatures (30°C) led to decrease in the acidity levels of the juice.

According to Table 1, the contents of total soluble sugars and reducing sugars increased during the storage period. The total soluble sugars were influenced by the interaction between treatments and storage periods (p < 0.05). Control fruits showed higher values of total soluble sugars. ‘Valencia Delta’ orange showed mean value of 7.02% in total soluble sugars.

The reducing sugars increased during the storage period, except in the control treatment, and showed significant differences (p < 0.05) during the storage period. The reducing sugars showed mean values of 3.14%. These values were higher than those observed by Hojo et al. (2010), who evaluated ‘Flame’ pomelos and obtained values between 2.62 and 1.19% of reducing sugars during 40 days of storage.

The content of ascorbic acid (Table 1) exhibited a reduction with significant difference (p < 0.05) during the days of storage, except in the control and coating treatments. In this experiment, the cv. ‘Valencia Delta’ showed mean values of ascorbic acid of 37.98 mg 100 g⁻¹, which is close to those

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Table 1. Titratable acidity, total soluble sugars, reducing sugars, ascorbic acid and total polyphenols in ‘Valencia Delta’ orange stored under refrigeration (7 °C; 85% RH)

<table>
<thead>
<tr>
<th>Treat.</th>
<th>0</th>
<th>7</th>
<th>14</th>
<th>21</th>
<th>28</th>
<th>35</th>
</tr>
</thead>
<tbody>
<tr>
<td>RD</td>
<td>0.57 aA</td>
<td>0.52 aA</td>
<td>0.58 aA</td>
<td>0.64 aA</td>
<td>0.60 aAB</td>
<td>0.47 aA</td>
</tr>
<tr>
<td>D</td>
<td>0.57 aA</td>
<td>0.52 aA</td>
<td>0.54 aA</td>
<td>0.60 aA</td>
<td>0.54 aA</td>
<td>0.54 aA</td>
</tr>
<tr>
<td>R</td>
<td>0.57 aA</td>
<td>0.66 aA</td>
<td>0.60 aA</td>
<td>0.72 aA</td>
<td>0.70 bA</td>
<td>0.55 aA</td>
</tr>
<tr>
<td>C</td>
<td>0.57 aA</td>
<td>0.58 aA</td>
<td>0.62 aA</td>
<td>0.68 aA</td>
<td>0.63 aAB</td>
<td>0.54 aA</td>
</tr>
</tbody>
</table>

- **Total soluble sugars (%)**
  - RD: 6.54 aA, 6.98 bcB, 6.94 bcA, 6.79 BA, 6.84 BB, 7.14 cB
  - D: 6.54 aA, 6.71 aBA, 7.01 cA, 7.23 DB, 6.68 abAB, 6.87 bcA
  - R: 6.54 aA, 7.10 cB, 7.48 dB, 6.89 BA, 6.58 Aa, 7.24 cB
  - C: 6.54 aA, 7.78 aBC, 7.45 cB, 7.66 cdC, 7.08 BC, 7.77 dC

- **Reducing sugars (%)**
  - RD: 2.83 aA, 3.36 aBa, 4.13 baA, 3.09 aA, 3.22 abA, 3.72 abA
  - D: 2.83 aBa, 3.24 aBa, 3.62 bA, 3.04 abA, 3.27 aA, 3.19 aA
  - R: 2.83 aA, 2.92 aA, 3.58 aA, 3.13 aA, 3.46 aA, 2.56 aA
  - C: 2.83 aBa, 3.73 aAa, 4.27 baA, 3.59 abA, 3.02 aA, 3.48 abA

- **Ascorbic acid (mg ascorbic acid 100 g⁻¹ of juice)**
  - RD: 42.50 b, 38.02 ab, 32.72 ab, 33.03 ab, 33.02 ab, 30.49 a
  - D: 42.50 b, 36.16 aBa, 36.32 ab, 40.21 ab, 34.52 a, 34.72 a
  - R: 42.50 a, 33.92 a, 34.92 a, 34.40 a, 34.48 a, 39.21 a
  - C: 42.50 a, 35.04 a, 36.09 a, 35.47 a, 32.29 a, 35.68 a

- **Total polyphenols (mg GAE 100 mL⁻¹)**
  - RD: 41.81 aBa, 41.68 abA, 40.26 abA, 41.91 aBa, 41.78 aBa, 45.41 bA
  - D: 41.81 aBa, 38.08 aBa, 46.68 bB, 42.72 aBa, 45.90 bAb, 45.20 bA
  - R: 41.81 aBa, 39.10 aBa, 43.01 abAB, 43.41 abA, 46.71 bB, 42.94 abA
  - C: 41.81 abcA, 37.25 aA, 43.44 bcbAB, 40.37 abA, 46.78 bB, 46.14 cA

1Means followed by the same letter, lowercase in row or uppercase in column, do not differ by Tukey test (p < 0.05); RD – Coating after degreening; D – Degreening; R – Coating; C – Control

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Figure 4. Luminosity in the analysis of peel color ‘Valencia Delta’ orange stored under refrigeration (7 °C; 85% RH)

Figure 5. Total chlorophyll in the peel of ‘Valencia Delta’ orange stored under refrigeration (7 °C; 85% RH)
reported by Pereira et al. (2013), who obtained mean of 37.53 mg 100 g⁻¹ for refrigerated 'Valência Delta' orange.

Mayuoni et al. (2011) also observed reduction in the contents of ascorbic acid in 'Navel' orange after three days at 20 °C; however, this reduction was observed in both treatments (control fruits and degreened fruits) and, therefore, was not attributed to the exposure to ethylene. On the other hand, in 'Star Ruby' pomelo and 'Satsuma' mandarin, there was no reduction in the levels of ascorbic acid, according to Mayuoni et al. (2011).

The contents of total polyphenols showed increments during the storage (Table 1). There was significant difference (p < 0.05) between the treatments and the storage period. The mean content of total polyphenols was 42.75 mg GAE 100 mL⁻¹. Gardner et al. (2000), evaluating samples of fruit juices, observed values of 50.4 mg GAE 100 mL⁻¹ for juice of 'Flórida' orange. Mayuoni et al. (2011) did not observe changes in the content of phenolic compounds in degreened 'Navel' orange.

**Conclusions**

1. The exposure of 'Valência Delta' orange to exogenous ethylene, at concentration of 5 to 10 ppm of ethylene gas (C₂H₄), promoted the degreening of the fruits.
2. The application of carnaúba-based wax preserved the external quality and physicochemical properties of 'Valência Delta' orange stored under refrigeration.
3. The use of carnaúba-based wax coating and refrigerated storage were fundamental for the conservation of quality in 'Valência Delta' orange after degreening, avoiding greater weight loss of the fruits.

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**Literature Cited**


