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Quality of 'Valencia Delta' orange after degreening and coating with wax

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Key words:
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exogenous ethylene
coating
storage

ABSTRACT
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.

Palavras-chave:
pós-colheita
etileno exógeno
revestimento
armazenamento

Qualidade de laranja 'Valência Delta' após desverdecimento e recobrimento com cera

RESUMO
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 °C) durante 35 dias; as variáveis analisadas incluíram perda de massa, coloração da casca, acidez titulável, ácido ascórbico, açúcares solúveis totais e redutores, polifenois 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 polifenois 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.

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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 Methds

Fruits of 'Valência Delta' orange [*Citrus sinensis* (L.) Osbeck], 'Swingle' citrumelo 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, removing any non-uniformity with respect to form, size, color, mechanical injuries or physiological damages.

The fruits were separated into four equal lots; lots 1 and 2 received 200 mL of concentrated ethyl solution to obtain 5 to 10 ppm of ethylene gas (C_2H_4), for the degreening process. The treatment with exogenous ethylene was applied in a refrigeration chamber at 30 ± 2 ºC and 80 ± 5% RH for 48 h, with intervals of 12 h for the renewal of the air in the chamber and consequent removal of the accumulated CO_2. The concentration of ethylene in the chamber was performed through an ethylene generator (Model 8014-400A, Matheson-Kitagawa).

At the end of this process, the fruits of lot 1 were coated (Aruá Tropical® – 50%, diluted in distilled water) and codified as RD. The fruits subjected to degreening and without coating were codified as D. The third lot of fruits was subjected only to coating (R) and the lot 4 was the control (C).

After the application of the treatments, the fruits were stored under refrigeration (7 ± 2 ºC and 85 ± 2% RH); the analyses were performed immediately after harvest and every seven days for a period of 35 days of storage.

Two experiments were carried out simultaneously, one for the non-destructive analyses (weight loss and external color) and another for destructive analyses (physical and physicochemical analyses). Non-destructive analyses had 20 replicates, while destructive analyses had 4 replicates.

Weight loss (previously numbered fruits) was calculated based on the differences in weight of the experimental units observed from experiment installation until the evaluation during storage, using a scale, with results expressed in percentage (%). The color of the external surface of the fruit was determined with two readings on opposite sides (previously marked) in the equatorial region of the fruits. Readings were performed using a Minolta CR-300 colorimeter, obtaining the value of L (luminosity), a* (red to green) and b* (yellow to blue). The values of chroma and hue angle were later calculated according to McGuire (1992).

Titratable acidity 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^{-1} of juice). The total soluble sugars were determined according to the methodology described by Yemn & 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 the Folin-Ciocalteau reagent, using gallic acid as the standard, according to the methodology described by Bucic-Kojic et al. (2007). Readings were performed in spectrophotometer at 765 nm and the results were expressed in mg GAE 100 mL^{-1}. Total chlorophyll content was evaluated by weighing 2.0 g of sample, which were then ground and mixed with 18 mL of 80% acetone. The mixture was filtered in a beaker wrapped in aluminum paper; the reading of the supernatant was performed in spectrophotometer at wavelength of 663 nm (chlorophyll a) and 646 nm (chlorophyll b). The results were expressed in mg 100 g^{-1}, calculated through the equations of Lichtenthaler (1987): Chlorophyll a (Ca) = $12.25 \times A_{663} - 2.79 \times A_{646}$; Chlorophyll b (Cb) = $21.50 \times A_{646} - 5.10 \times A_{663}$; Total chlorophyll (Ct) = $7.15 \times A_{663} + 18.71 \times A_{646}$.

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 3º degree and the minimum value of R^2 was 70%.

Results and Discussion

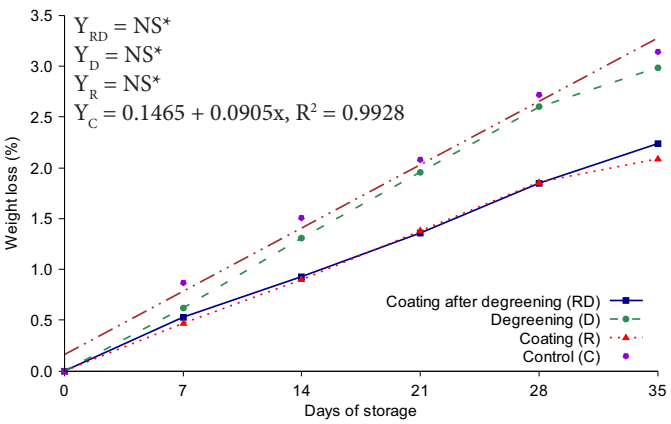
The use of coating in 'Valência Delta' orange showed relevant effect, since it reduced the weight loss and promoted better visual quality in the fruits during storage. According to Figure 1, the weight loss of 'Valência Delta' orange increased during the storage in all treatments; however, there was 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



NS* Not significant

Figure 1. Weight loss in 'Valência Delta' orange stored under refrigeration (7 °C; 85% RH)

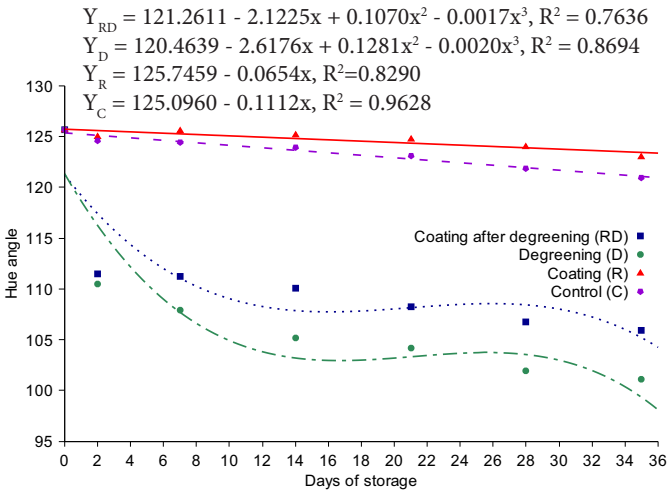


Figure 2. Hue angle of peel color in 'Valência Delta' orange stored under refrigeration (7 °C; 85% RH)

from 124 to 100, analyzing 'Lane late' oranges subjected to the degreening treatment.

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.

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

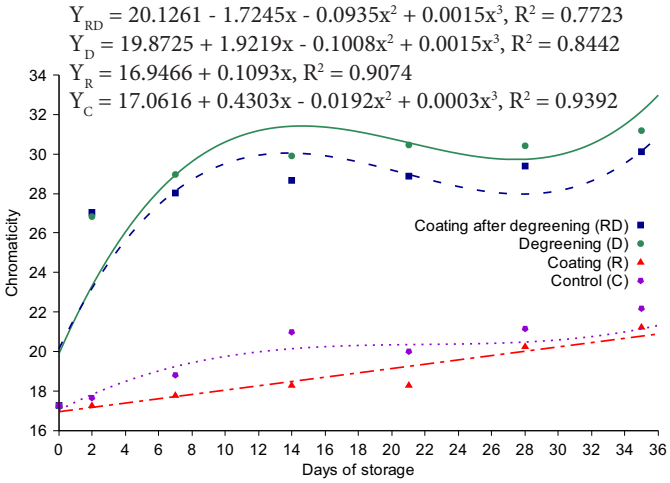


Figure 3. Chromaticity in the analysis of peel color in 'Valência Delta' orange stored under refrigeration (7 °C; 85% RH)

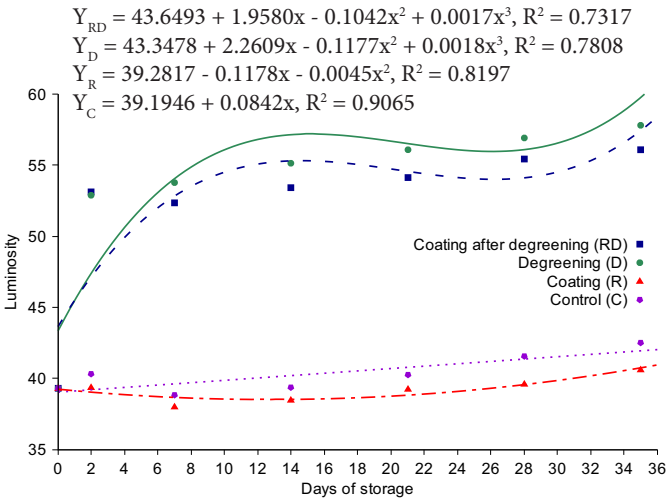


Figure 4. Luminosity in the analysis of peel color 'Valência Delta' orange stored under refrigeration (7 °C; 85% RH)

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. 'Valência Delta' orange showed acidity around 0.6%. It was observed that the values remained statistically equal along the storage period in all treatments. Rapisarda et

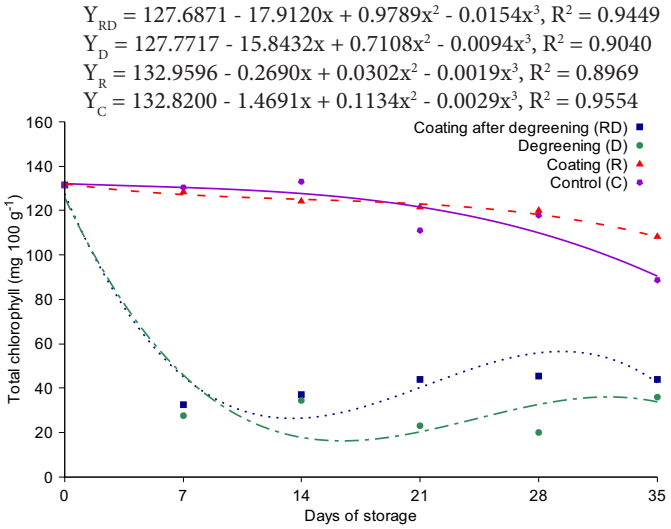


Figure 5. Total chlorophyll in the peel of 'Valência Delta' orange stored under refrigeration (7 °C; 85% RH)

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

Treat.	Days of storage					
	0	7	14	21	28	35
Titratable acidity (% citric acid)						
RD	0.57 aA	0.52 aA	0.58 aA	0.64 aA	0.60 aAB	0.47 aA
D	0.57 aA	0.52 aA	0.54 aA	0.60 aA	0.54 aA	0.54 aA
R	0.57 aA	0.66 aA	0.60 aA	0.72 aA	0.70 aB	0.55 aA
C	0.57 aA	0.58 aA	0.62 aA	0.68 aA	0.63 aAB	0.54 aA
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 dC	7.45 cB	7.66 cdC	7.08 bC	7.77 dC
Reducing sugars (%)						
RD	2.83 aA	3.36 abA	4.13 bA	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 abA
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 abA	4.27 bA	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 ab	38.32 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 aA	41.91 abA	41.78 abA	45.41 bA
D	41.81 abA	38.08 aA	46.68 bB	42.72 abA	45.90 bAB	45.20 bA
R	41.81 abA	39.10 aA	43.01 abAB	43.41 abA	46.71 bB	42.94 abA
C	41.81 abcA	37.25 aA	43.44 bcdAB	40.37 abA	46.78 dB	46.14 cdA

[1]Means 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

al. (2008) observed that, under different climatic conditions, there was increase in the contents of citric acid in the variety 'Valência', 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. 'Valência Delta' orange showed mean value of 7.02% in total soluble sugars.

The reducing sugars increased during the storage period, except in the coating treatment, and showed significant difference ($p < 0.05$) 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. 'Valência Delta' showed mean values of ascorbic acid of 37.98 mg 100 g^{-1}, which is close to those

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 ºC; 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 (C_2H_4), 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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