

Edible coatings on the post-harvest conservation of orange 'Natal CNPMF 112'

Revestimentos comestíveis na conservação pós-colheita da laranja 'Natal CNPMF 112'

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Highlights

The acidity and maturity index of the orange are affected by edible coatings.

The addition of carnauba wax to the orange decreases its acidity.

The application of alcoholic propolis to fruit causes reduced weight loss.

To protect the quality of the orange, alcoholic propolis should be applied.

Abstract

Citrus fruit have a lengthy post-harvest lifetime, however owing to water loss, they lose firmness and luster on the skin after being picked. In light of the foregoing, this study aimed to assess the effects of coatings on the post-harvest conservation of 'Natal CNPMF 112' oranges under refrigerated settings using aqueous and alcoholic propolis extracts and carnauba wax. Fruit of 'Natal CNPMF 112' orange tree grafted on 'Rangpur' lime were treated with carnauba wax (100%) and alcoholic and aqueous propolis extracts (30%). The experimental design was totally randomized, with the plots consisting of the coatings plus the control (distilled water), and the subplots separated by the storage time (0, 10, 20, and 30 days) at 12 °C ± 2, with the exception of weight loss, which was assessed at three-day intervals (0 to 30 days). Loss of fruit mass, average fruit mass, soluble solids, titratable acidity, ascorbic acid, maturity index, pH, and technical index were all examined. According to the findings, the alcoholic extract of propolis produced decreased fruit mass loss, a high maturity index, and balanced acidity. The coatings used in the postharvest conservation of refrigerated 'Natal CNPMF 112' oranges had no effect on the other parameters. As a result, propolis alcoholic extract is suggested for the preservation of 'Natal CNPMF 112' orange.

Key words: Storage. Modified atmosphere. Vegetable wax. *Citrus sinensis* (L.) Osbeck. Propolis.

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Resumo

Os frutos cítricos apresentam prolongado período pós-colheita, no entanto, devido à perda de água, após colhidos, perdem a firmeza e ficam com pouco brilho na casca. Face ao exposto, objetivou-se neste trabalho determinar os efeitos dos revestimentos na conservação pós-colheita da laranja 'Natal CNPMF 112' com utilização de extratos de própolis aquoso e alcoólico e a cera de carnaúba em condição refrigerada. Frutos da laranjeira 'Natal CNPMF 112', enxertada em limoeiro 'Cravo', foram tratados com cera de carnaúba (100%) e extratos de própolis alcoólico e aquoso (30%). O delineamento experimental foi inteiramente casualizado, com arranjo em parcelas subdivididas, sendo as parcelas constituídas pelos revestimentos mais a testemunha (água destilada), e as subparcelas pelo período de armazenamento (0, 10, 20 e 30 dias) a $12\text{ °C} \pm 2$, com exceção para perda de massa, a qual foi avaliada a intervalos de três dias (0 a 30 dias). Avaliaram-se: perda de massa dos frutos, massa média dos frutos, sólidos solúveis, acidez titulável, ácido ascórbico, índice de maturação, pH e índice tecnológico. Conforme os resultados, observou-se que o extrato alcoólico de própolis promoveu menor perda de massa do fruto, bom índice de maturação e acidez equilibrada. As demais características não foram afetadas pelos revestimentos utilizados na conservação pós-colheita da laranja 'Natal CNPMF 112' refrigerada. Portanto, recomenda-se o extrato alcoólico de própolis para a conservação da laranja 'Natal CNPMF 112'.

Palavras-chave: Armazenamento. Atmosfera modificada. Cera vegetal. *Citrus sinensis* (L.) Osbeck. Própolis.

Introduction

Citrus cultivation has encountered significant problems in recent years. Nevertheless, in 2021, the citrus belts of São Paulo and Triângulo/Southwest Mineiro projected 294.17 million boxes of oranges for the year 2021/2022. Brazil produces 34% of all oranges and more than half of all juice produced worldwide, accounting for 76% of global orange juice commerce (Fundo de Defesa da Citricultura [FUNDECITRUS], 2022). There has been a surge in orange groves in the Northeast region in recent years, which has particular climatic features and a fortunate geographical position, such that, if carefully utilized, it might stand out as the key area for citrus expansion in Brazil (Passos et al., 2011).

The sweet orange cultivars Pera, Valência, Natal, and Folha Murcha are the most well-known, sweet orange trees grown

in Brazilian citrus industry (Bastos et al., 2015). Several oranges, such as Embrapa's 'Natal CNPMF 112', grown from a nucellar clone of the 'Natal' orange tree, have been introduced as an alternative. This orange tree is distinguished by its late maturing fruit and excellent output (40 t ha^{-1}). Ripe fruit are medium-sized (190 g), have a somewhat rough and uniform yellow peel, just four seeds per fruit, vivid orange flesh, and a juice level of around 57%, and are designated for the fresh fruit market or industrial (Empresa Brasileira de Pesquisa Agropecuária [EMBRAPA], 2019).

The customer requires certain qualitative qualities in the marketing of fruit, such as appearance, flavour, and nutritional content. In terms of post-harvest, organic acids, soluble solids, and pH levels are added. The major sources of quality loss during storage are physical and physicochemical changes, which are

influenced by factors like as harvest season, location, variety, agricultural practices, and post-harvest treatment (Neves, 2009). As a result of the expansion and establishment of new production regions, alongside the introduction of new cultivars, it's indeed vital to attempt to enhance the techniques used across the whole production chain, including those used in post-harvest.

Oranges, like other non-climacteric fruit, have a lowered respiratory rate after harvesting, which is sufficient to continue the transpiration process, resulting in water loss and, as a result, diminished brightness (Chitarra & Chitarra, 2005). Given the foregoing, several options, such as the application of edible coatings, have gained traction in recent years, owing primarily to the possibility for post-harvest conservation of diverse plant products (Santos et al., 2015).

The coatings applied to the fruit work as a protective barrier and, depending on their composition and thickness, have varying rates of permeability to O_2 , CO_2 , and water vapour, encouraging what is known as a modified environment on the surface of the fruit. Reduced O_2 levels and increased CO_2 levels result in reduced respiratory rates, especially when combined with cooling, and enhance the product's shelf life during storage (Chitarra & Chitarra, 2005). Carnauba wax is one of the most utilized coatings in fruit post-harvest. Other products, such as those made of starch, cellulose, gums, caseins, and propolis, can be utilized instead.

In light of the foregoing, the goal of this study was to assess the effects of edible coatings on the post-harvest conservation of 'Natal CNPMF 112' oranges under refrigerated circumstances using aqueous and alcoholic propolis extracts and carnauba wax.

Material and Methods

The research was conducted in the Chemistry Laboratory of the Serra Talhada Academic Unit/ Federal Rural University of Pernambuco in Serra Talhada, Pernambuco, Brazil. Fruit of 'Natal CNPMF 112' sweet orange trees grafted on 'Rangpur' lime from the Active Citrus Germplasm Bank of Embrapa Semi-Arid, Bebedouro, Petrolina, PE. The fruit were collected as they reached maturity for the fresh fruit market. They were transported to the laboratory, selected by size and color pattern, rinsed in running water, sanitized in a sodium hypochlorite solution (2.2 mg L^{-1} active chlorine) for 15 min, and dried in a dry condition.

Carnauba wax (100%) and alcoholic and aqueous propolis extracts (30%) were applied to the fruit, along with distilled water (control). The Química JVC Ltda Co. from Feira de Santana, Bahia, Brazil, supplied the pure commercial product (100%) JVC WAX - 36 A (made of 36% carnauba wax). The aqueous propolis extract used was extracted from the commercial product Propolis Extract (composed of 30% dry propolis and deionized water) by the company APIS FLORA Indústria e Comercio Ltda, Ribeiro Preto, Sao Paulo, Brazil, and the alcoholic propolis extract was extracted from the product Commercial Alcoholic Propolis Extract (30% propolis and neutral alcohol) by the same company APIS FLORA.

The products were applied manually, and the dried fruit were kept in refrigerated settings with relative humidity between 90 and 95%. The experimental design consisted of completely randomized blocks arranged in subdivided plots, with the plots consisting of coatings (control, carnauba wax, and

alcoholic and aqueous extracts of propolis) and the subplots by storage time (0, 10, 20, and 30 days), except for weight loss, which was evaluated at three-day intervals (0 to 30 days). We utilized four repetitions of 20 fruit per plot. Physical and physicochemical analyses of the fruit were used to assess the impact of the coatings. The juice yield (RS) was calculated as a percentage using the equation $RS = \text{juice mass} / \text{total fruit weight} \times 100$. A digital refractometer was used to determine the soluble solids (SS) content, and the readings were expressed in °Brix. Titration with 0.1N NaOH was used to determine the titratable acidity (TA) in a solution of 5 mL of juice and 95 mL of distilled water, with the result represented as a percentage of citric acid (Instituto Adolfo Lutz [IAL], 2008). The maturation index was calculated using the link between soluble solids and titratable acidity. Titration with iodine (1%), with starch (1%) as an indicator, yielded the ascorbic acid concentration, reported in $\text{g } 100 \text{ g}^{-1}$. (S. L. A. Silva et al., 1995). A pH meter was used to measure the pH of the homogenized juice. Ultimately, the technological index was calculated using the formula $SS \times \text{pulp yield} / 100$ (Chitarra & Chitarra, 2005).

The weight loss of the fruit was also assessed. For this purpose, the fruit were weighed at three-day intervals (0 to 30 days)

and the difference in weight between the intervals was calculated, expressed as a percentage, using the equation: $\% \text{ loss of mass} = ((MI - MA / MI) \times 100)$, where MI is the initial mass (day zero) and MA is the mass on the day of the assessment.

The acquired data were subjected to analysis of variance, and when significant, the Tukey Test was used for the plots (coatings) and the regression test for the subplots (storage time) at a 5% probability level.

Results and Discussion

According to the analysis of variance data, there appears to be a significant interaction ($p < 0.01$) between the applied edible coatings and the storage duration for weight loss of the 'Natal CNPMF 112' orange fruit. Regardless of the treatments used, fruit mass loss rose linearly over the refrigerated storage period (Figure 1). Weight loss is directly connected to metabolic activities like as transpiration and fruit respiration, which are responsible for diminishing the quantity of water available in plant tissue. As a result, water loss occurs not only in quantitative losses, but also in qualitative losses, such as nutritional, textural, and visual features (Chitarra & Chitarra, 2005).

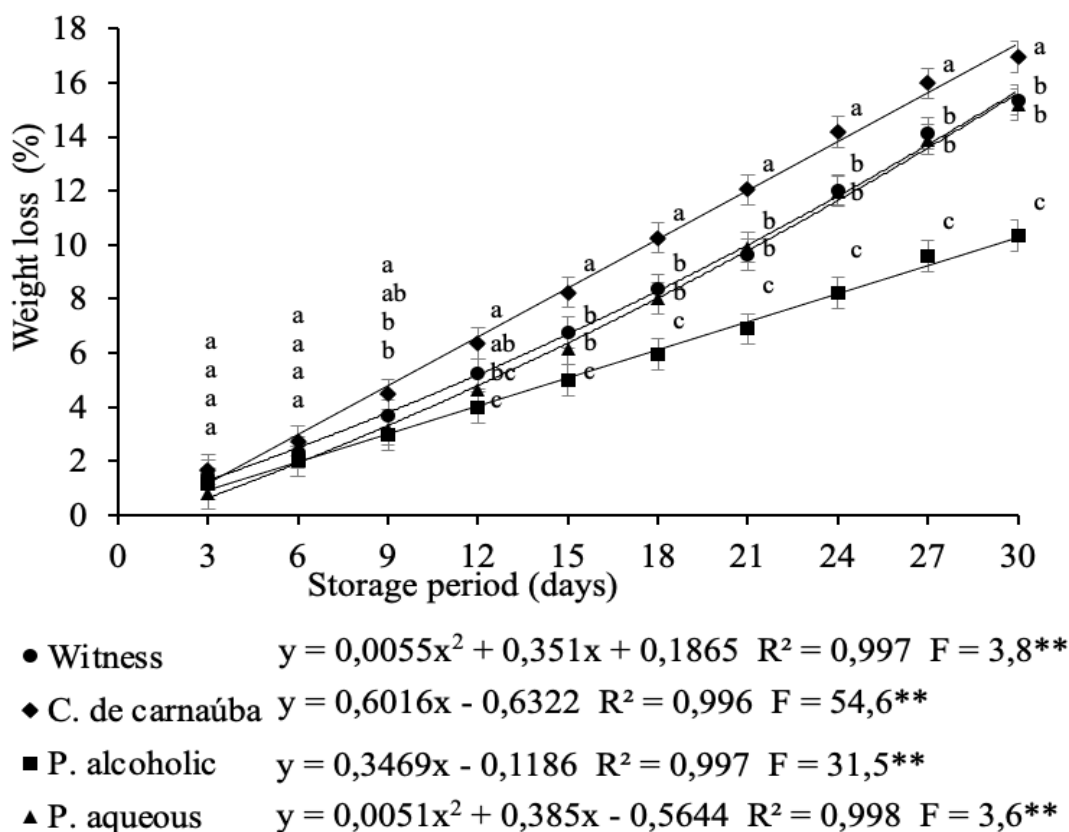


Figure 1. Weight loss of 'Natal CNPMF 112' orange subjected to various postharvest treatments and refrigerated storage period at $12^{\circ}\text{C} \pm 2$. Serra Talhada, PE. 2021. Bars = the least significant difference (DMS).

In the current investigation, there was no significant difference in weight loss between fruit treated with various coatings under refrigerated storage settings until the sixth day following treatment application. Nevertheless, the fruit treated with a coating based on carnauba wax exhibited the largest mass loss, with 16.97%, which was substantially greater in comparison to the coatings with propolis extracts, as of the ninth day following application of the products. The fruit of the control and alcoholic propolis treatments maintained intermediate values for loss of fruit mass without substantially

varying, reaching averages of mass loss of 15.37 and 15.19%, respectively, 30 days following application of the treatments (Figure 1).

Of the items tested, the alcoholic propolis extract resulted in the lowest mass loss, lasting from the 12th day to the end of storage, when the fruit lost 10.34% of their weight (Figure 1). Pereira et al. (2014) discovered a similar outcome when applying carnauba wax on 'Valencia Delta' oranges, with a fruit mass loss of 14% after 28 days of storage.

Ataíde et al. (2017) found that utilizing aqueous propolis in juazeiro fruit resulted in better weight reduction than treatments with alcoholic propolis and carnauba wax. Santos et al. (2015) observed lesser mass loss of avocado fruit when using carnauba wax, but greater fruit conservation when using both carnauba wax and alcoholic propolis (30%). The findings underscore the significance of additional post-harvest investigations in fruit, since various coatings might provide varied results.

Citrus fruit with mass losses more than 6% are unsuitable for commercialization because of drying of the skin and wrinkling of the fruit (Ladaniya, 2008). As a result, among the items used, alcoholic propolis kept the fruit of the 'Natal CNPMF 112' orange tree for a longer period than the others. Nevertheless, the mass loss was 6% towards the 18th day of storage (Figure 1).

Carnauba wax, propolis, paraffin, vegetable and mineral oils, and other edible coatings are primarily composed of polysaccharides, lipids, and proteins. Such materials encourage the production of a modified environment on the surface of the fruit, allowing edible coatings to replace plastic ones. As a result, a semipermeable barrier to water vapor and gases is formed. They also allow for the introduction of chemicals like as antioxidants and antimicrobials, which are vital in maintaining the mechanical integrity of plants (Chitarra & Chitarra, 2005; Botrel et al., 2010).

There was no significant interaction between coatings and storage duration for the juice production of the 'Natal CNPMF 112' orange. There is a quadratic increase in the means up to the 24th day of refrigerated storage, followed by a decline in the average juice yield values up to the final period of storage (30 days) (Figure 2).

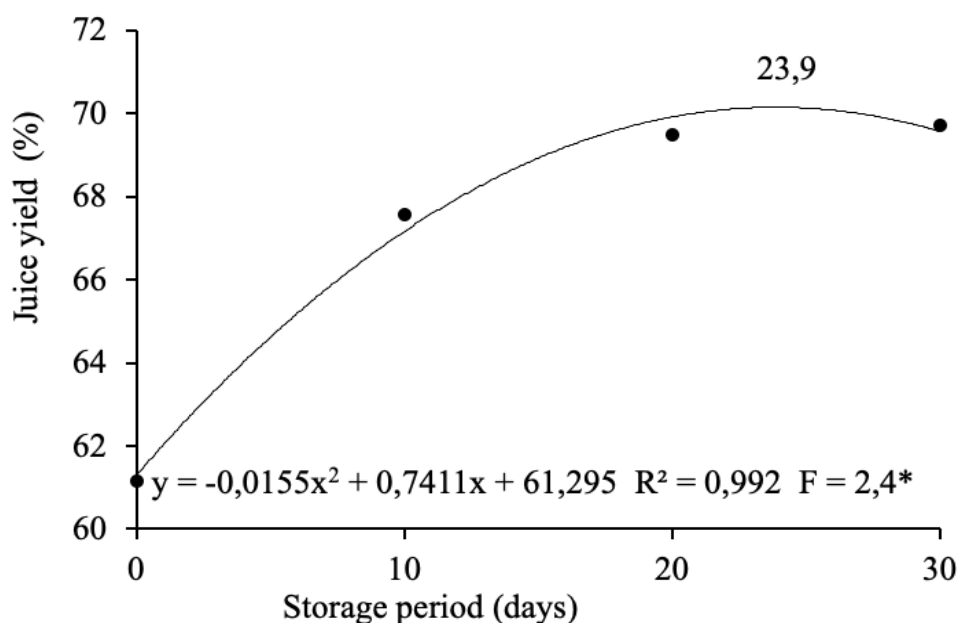


Figure 2. 'Natal CNPMF 112' orange juice yield subjected to various post-harvest treatments and refrigerated storage period at 12°C ± 2. Serra Talhada, PE. 2021.

Citrus juice yields should be more than 35%, since the industry demands a minimum value of 40%. (Koller, 1994). The percentage of juice yield attained for the 'Natal CNPMF 112' orange in the current study was higher than that needed by the industry, with an average juice yield of 69.70%. (Figure 2). While Beber et al. (2018) reported quadratic behaviour for juice yield in sweet oranges under Rio Branco/Acre conditions, the average juice yield was lower (45%) than the average obtained in the current study. Miranda and Campelo (2012)

found a similar finding, with an average yield value for the 'Pêra' orange ranging from 39.34 to 48.71%.

There was a significant interaction between coatings and storage for titratable acidity ($p < 0.01$) and maturity index ($p < 0.01$) when analysing the physicochemical parameters of the juice. Only storage duration had a significant influence on soluble solids, pH, technological index, and ascorbic acid ($p < 0.05$) (Table 1).

Table 1

F values from the analysis of variance, means and coefficients of variation (CV) of soluble solids (SS), pH, titratable acidity (AT), maturation index (MI), technological index (TI) and ascorbic acid (AA) of 'Natal CNPMF 112' orange subjected to different postharvest treatments and refrigerated storage period at 12°C ±2. Serra Talhada-PE. 2021

FV	GL	SS (°Brix)	pH	AT (% ác. cítrico)	IM (SS/AT)	IT	AA
Products (A)	3	1.2ns	2.6ns	1.1ns	0.8ns	1.5ns	2.7ns
Storage time (B)	3	18.9**	37.7**	1.2ns	12.8**	20.7**	4.6**
AxB	9	0.3ns	1.0ns	3.6**	4.5**	0.4ns	1.0ns
Means		12.53	3.74	0.77	15.60	8.39	17.75
CV1(%)		4.69	2.03	7.61	7.84	9.26	4.65
CV2(%)		6.46	1.89	5.40	6.05	9.61	5.67

NS = not significant; * = significant at 5 %; ** = significant at 1 % by the F test.

Until the 10th day of refrigerated storage, the titratable acidity of 'Natal CNPMF 112' orange fruit did not change substantially across edible coatings. However, 20 days after treatment, the control fruit (distilled water) had a greater titratable acidity content, which did not vary from those treated with propolis alcoholic extract and carnauba wax. At 30 days, the fruit treated with aqueous

propolis had the maximum acidity, which did not differ from the control or the alcoholic extract of propolis (Figure 3). Fruit treated with aqueous propolis extract, on the other hand, showed a quadratic reduction in titratable acidity up to the 13th day, followed by a rise in acidity up to 30 days following treatment application (Figure 3).

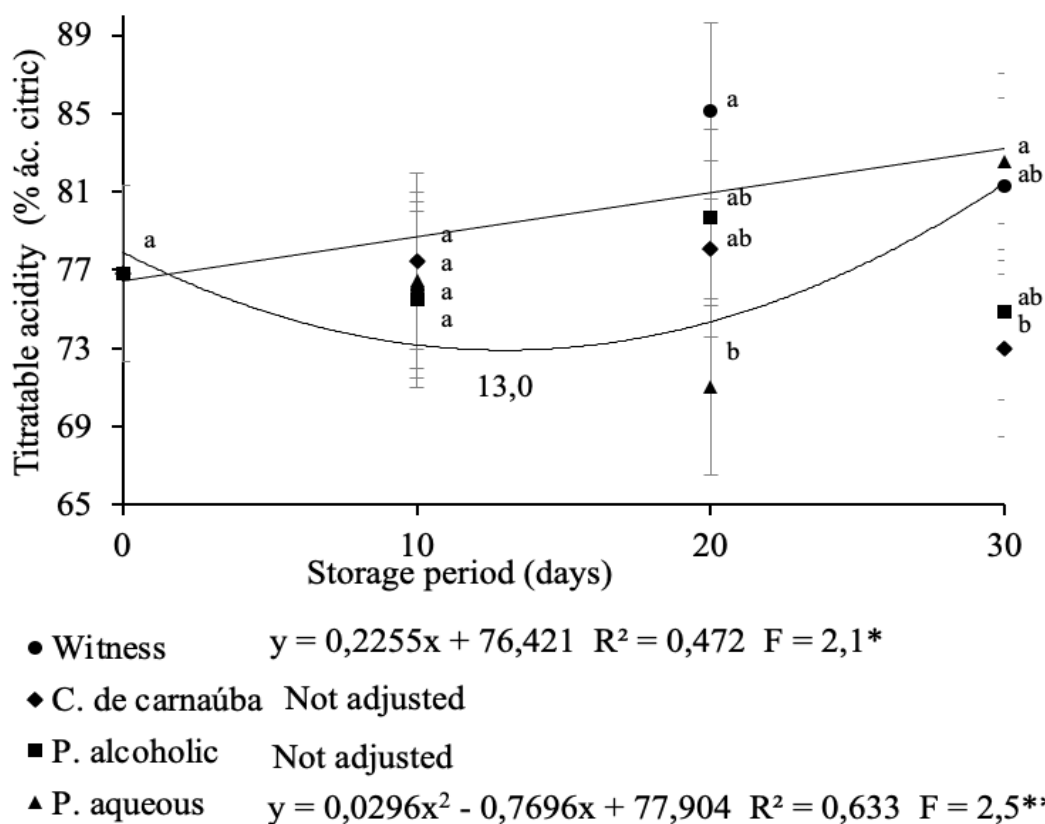


Figure 3. Titratable acidity of "Natal CNPMF 112" orange subjected to various post-harvest treatments and refrigerated storage period at 12°C ± 2. Serra Talhada-PE. 2021. Bars = the least significant difference (DMS).

To avoid an increase in the fruit's insipid flavour, the acidity must be too low, especially when meant for eating, with criteria set below 0.9% and above 0.6%. (Pozzan & Triboni, 2005). The results of this study are within the acceptable range, with averages ranging from 0.774 to 0.825% of titratable acidity. Beber et al. (2018) achieved similar acidity levels after 228 days of maturity in four progenies of sweet orange trees grafted onto Rangpur lime (*Citrus limonia* Osbeck), with acidity values ranging from 0.57 to 0.69%. Portela et al. (2006) found 0.42% titratable acidity in 'Pêra' orange.

When the treatments with varying coatings of 'Natal CNPMF 112' orange were tested as a function of refrigerated storage duration, it was found that the titratable acidity of the fruit of the control treatment rose linearly (Figure 3). The authors Ataíde et al. (2017) obtained the rise in acidity seen in that work in a post-harvest investigation with juazeiro fruit, and Passos et al. (2014) analyzed it in an experiment with oranges. Santos et al. (2015) discovered different findings in an experiment with avocado employing edible coatings, with mean titratable acidity dropping linearly.

Orange juice comprises water, sugars, organic acids, phenolic compounds, which are among the primary constituents of citrus fruit, as well as pectin, fiber, and lipids. The organic acid is a significant qualitative element of the fruit, impacting the flavour directly (Angelo & Jorge, 2007).

The increase in acidity is attributed to the release of galacturonic acids, causing an increase in fruit ripening by the action of the enzymes pectin methylesterase and polygalacturonase, attributed to the low respiratory metabolism and storage temperature, the measure in which there is acid accumulation in the vacuoles, resulting in lower consumption of organic acids, leading to an increase in soluble solids levels (Alves et al., 2000; Damiani et al., 2008). The increase in titratable acidity observed in the experiment can also be explained by the dilution factor, or the loss of water from the fruit during storage, which promotes the concentration of acids and sugars, when the orange is considered a non-climacteric fruit with a low respiration rate and ethylene production.

The maturation index of oranges treated with alcoholic extract of propolis increased linearly throughout the evaluation period, with intermediate values obtained at 10 and 20 days of refrigerated storage, reaching the highest average at 30 days of storage, though it did not differ significantly from the treatment with propolis aqueous extract and the control. The maturity index of fruit treated with carnauba wax and propolis aqueous extract increased quadratically up

to 16.4 and 19.0 days of storage, respectively (Figure 4). In research with juazeiro fruit, Ataíde et al. (2017) found a similar trend.

The sweet flavour of the fruit is determined by the ripeness index; the higher the ripeness index of the fruit, the greater the manifestation of the sweet taste. As a result, the orange treated with propolis alcoholic extract has a greater maturity index as well as less mass loss. The maturity index reported in this study is comparable to that obtained by Menezes and Brunini (2016), with values ranging from 12.23 to 19.3 for 'Pêra' oranges treated with wax and stored under refrigeration.

There was a minor quadratic decline in the average contents up to 10.2, followed by a strong increase up to 30 days of storage, with an average of 14.03 °Brix, independent of the treatment utilized (Figure 5A). The Brazilian Program for Horticultural Modernization (Companhia de Entrepósitos e Armazéns Gerais de São Paulo [CEAGESP], 2011) specifies a minimum soluble solids concentration of 10 °Brix for most orange types. The values obtained in the current investigation for the 'Natal CNPMF 112' orange were greater than the specified minimum (Figure 5A) and higher than the findings obtained by Pereira et al. (2014), who got 10.75 °Brix in 'Valencia Delta' orange using carnauba wax. Passos et al. (2014) discovered values ranging from 8.25 to 9.16 °Brix in 'Pêra' orange treated with propolis extract.

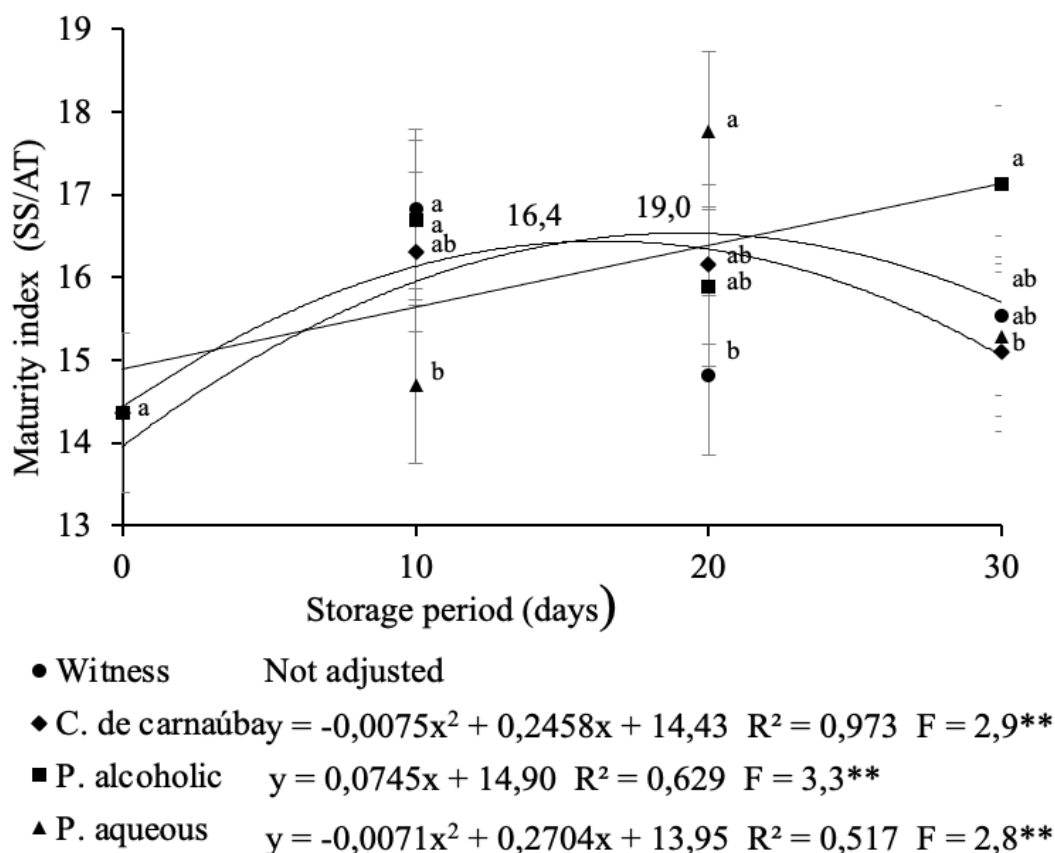


Figure 4. Maturity index of 'Natal CNPMF 112' orange subjected to various post-harvest treatments and refrigerated storage period at 12°C ± 2. Serra Talhada-PE. 2021. Bars = the least significant difference (DMS).

In terms of pH, the values dropped in a quadratic manner up to 28 days and stayed constant up to 30 days of storage (Figure 5B). This decrease is due to the formation of organic acids throughout the storage period (P. T. Silva et al., 2005). The mean pH value (3.77) recorded in this study appears to be lower than that found by Pereira et al. (2014), who reported a pH value of 4.37 in 'Valencia Delta' orange. P. S. Silva et al. (2017) used edible films to achieve a pH of 2.85 in 'Salustiana' oranges in a post-harvest investigation. Nevertheless, pH is an essential

characteristic in fruit destined for processing since it influences the appreciation of the juice and the condition of conservation of the final product (Oliveira et al., 1999).

The ascorbic acid content of fruit juices is found in the human body in the form of ascorbate, a water-soluble vitamin that plays a direct role in collagen synthesis while also acting as an antioxidant, reducing transition metals (Fe 3+ and Cu 2+) in iron absorption, and promoting the prevention and cure of colds (Duzzioni, 2010; Mahan & Escott, 2010).

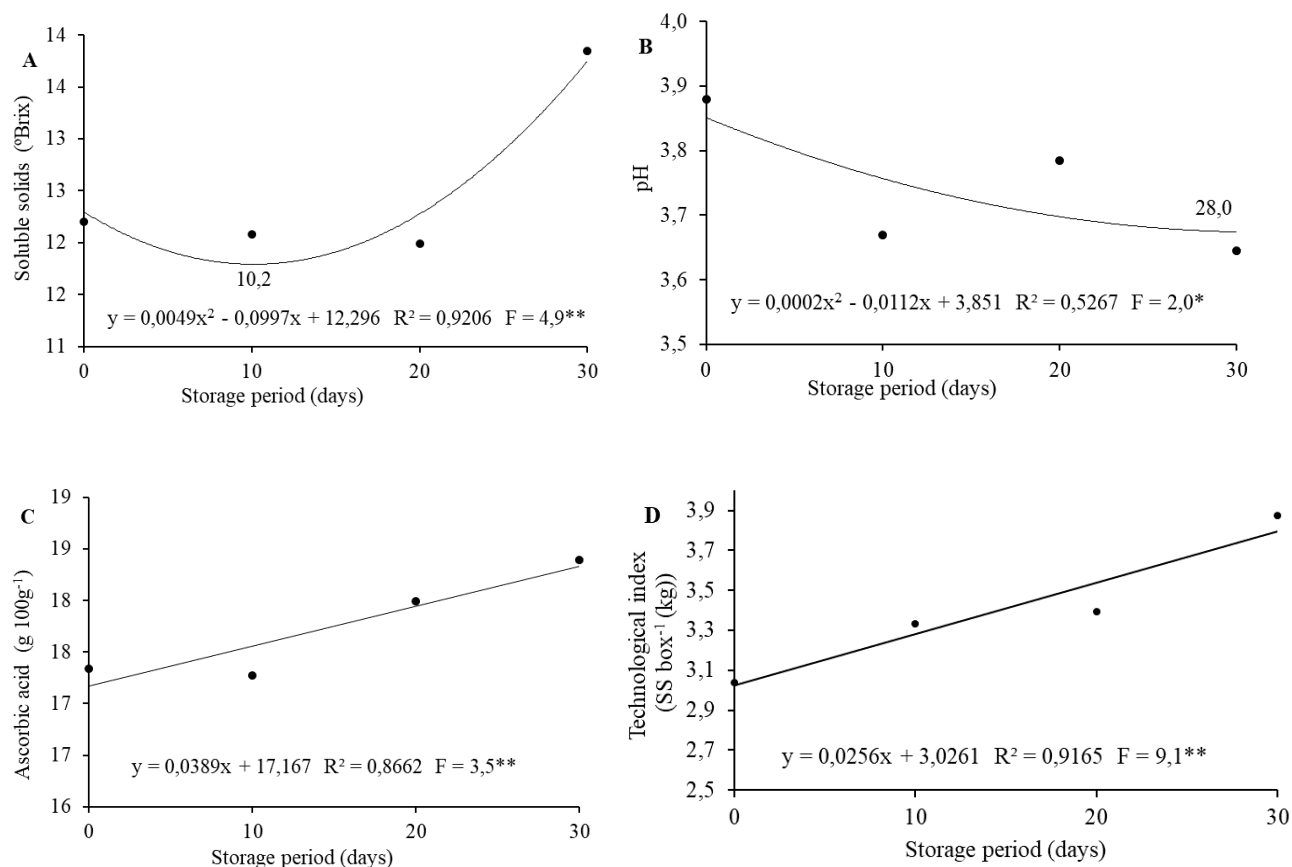


Figure 5. Soluble solids (A), pH (B), ascorbic acid (C), and technological index (D) of 'Natal CNPMF 112' orange after various post-harvest treatments and refrigerated storage period at 12°C ±2. Serra Talhada-PE. 2021.

In the current study, a linear rise in mean ascorbic acid concentration was found as storage duration increased (Figure 5C). Whereas investigations utilizing carnauba wax by Pereira et al. (2014) and cassava starch by Agostini et al. (2014) reported greater levels of ascorbic acid in oranges than the current research, with values of 37, 28 and 46.37, respectively.

In terms of the technological index or industrial yield of the fruit, a linear rise as a function of storage duration is observed, with an average value of 3.8 achieved (Figure 5D). This quality metric relates pulp yield to

soluble solids %, reflecting the yield of the end processing product. It takes into account fruit destined for industry with a higher technical index, meaning fruit with a greater sugar content in the pulp (Chitarra & Chitarra, 2005). Sombra et al. (2018) reported a higher value in 'Russian' orange in semi-arid settings (in the state of Ceará), with means ranging from 2.51 to 2.63. Tazima et al. (2009) observed an average value of 2.5 on 'Natal I-60'. The average recorded in 'Seleta- Vermelha I-17' orange from IAPAR's Active Germplasm Bank in Londrina, PR, Brazil, was 1.8.

Conclusions

The products used affect the titratable acidity and the maturation index of the fruit differently, so that at 30 days the 'CNPMF Natal 112' oranges covered with carnauba wax have lower acidity than the control fruit, while those covered with alcoholic propolis extract have a higher index of maturation.

In overall, the alcoholic propolis extract stands out for encouraging minimal mass loss, a high maturity index, and balanced acidity, despite the fact that the other attributes were unaffected by the products' use. Consequently, it is the most recommended coating among the assessed coatings for keeping the quality of the 'CNPMF Natal 112' oranges.

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