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RESEARCH ARTICLE

Ethylene absorber ($KMnO_4$) in postharvest quality of pinha (*Anona squamosa* L.)

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ABSTRACT

The ripening pattern of the climacteric type of the pinha (*Anona squamosa* L.) limits its shelf life at room temperature, in addition, storage at low temperatures develops cold sores on the fruit. Thus, the association of conservation technologies is fundamental to reduce the losses in the post-harvest of this fruit, so it was aimed to evaluate sachets impregnated with potassium permanganate ($KMnO_4$) associated with refrigeration in the control of ripening and preservation of quality. For this, pinhas were harvested at physiological maturity, packed in polystyrene styrofoam trays coated with PVC film with and without the presence of sachets containing 3 g of $KMnO_4$ and stored at 13 °C for 20 days. Every five days the fruit quality was evaluated as: weight loss, cracking index, firmness, external appearance, starch content, soluble solids, titratable acidity, pH, ratio SS/TA, coloring (*Hue*, Chroma and Luminosity) and cold damage. The absorption of ethylene by the $KMnO_4$ sachet inside the packages preserved significantly ($p < 0.05$) the physical-chemical quality and the visual appearance of the fruits but did not influence the coloration (chroma and luminosity) and incidence of damage by cold in relation to its control at the end of the storage period. The packing of pinhas containing 3 g of $KMnO_4$ is an alternative to delay ripening, prolong the shelf life without compromising the physical-chemical quality of the fruits.

Keywords: *Anona squamosa* L.; Physical-chemical quality; Potassium permanganate; Refrigeration

INTRODUCTION

The pinha, earlberry (*Annona squamosa* L.) is a fruit of tropical origin belonging to the Anonaceae family, originating in Central America (Trindade Island, Antilles) (Manica et al., 2003) and appreciated for its pleasant flavor and odor. Because it is a climacteric fruit, the rapid maturation after harvest compromises the shelf life in three to four days when kept at room temperature due to the rapid loss of firmness of the pulp, which is why it is marketed only in the domestic market (Gõni et al., 2010; Silva et al., 2013). The refrigeration is also limited due to the sensitivity of the anonacea species to low storage temperatures due to their tropical origin caused by cold sores altering the color of the bark and pulp (Silva and Muniz, 2011).

To reduce postharvest losses, it is essential to improve the technologies and know the factors involved in the deterioration of the fruits, as well as the techniques that delay senescence and preserve the quality of the fruits, guaranteeing food and nutritional security (Amarante and Steffens, 2009; Gustavsson et al., 2011). Ethylene (C_2H_4) is a compound released during the climacteric fruit metabolism, which activates and controls several physiological mechanisms, among which the maturation and senescence of climacteric fruits (Chitarra and Chitarra, 2005; Cappellin et al., 2014), through a series of reactions involving the degradation of chlorophyll and synthesis of pigments (carotenoids and flavonoids), modification of the texture by changes in the turgor and structure of the cell wall, changes in sugars, acids, and volatile profiles affecting nutritional quality (flavor and aroma), as well as increased susceptibility to opportunistic pathogens due to cell wall vulnerability (Klee and Giovannoni, 2011; Gapper et al., 2013; Gómez et al., 2014).

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The technique of ethylene absorption with the use of potassium permanganate impregnated sachets ($KMnO_4$) is an alternative in the reduction of the ethylene produced during maturation, prolonging the pre-climacteric phase and the post-harvest life of the fruits (Resende et al., 2001), because they absorb and oxidize ethylene to water, carbon dioxide, manganese dioxide and potassium (Wills and Warton, 2004; Wills et al., 2014) and because it is a volatile compound, it can be physically separated from the product, eliminating the risk of chemical injury (Sá et al., 2008).

The efficiency of $KMnO_4$ in the control of ripening has already been evaluated in fruits such as blueberry (Brackmann et al., 2010); kiwi (Bal e Celik, 2010); jaboticaba (Nascimento et al., 2013), mangaba (Nasser et al., 2015) banana (Wills et al., 2014; Falcão et al., 2017), abricó (Moradinezhad and Jahani, 2019) and considering the problems posed by the postharvest of pinha and the scarcity of work related to fruit storage, the objective of this study was to evaluate whether sachets with potassium permanganate are capable of delaying maturation preserving the physicochemical characteristics during 20 days storage at 13 ºC.

MATERIAL AND METHODS

Plant material

The 'Crioula' pinhas was harvested at the physiological maturity, at the beginning of the removal of carpels, in an orchard located in the municipality of Brasil Novo, PA situated under the geographical coordinates of 43º 47' 39,7" west longitude e 15o 05' 51,6" south latitude, with altitude of 452 m. The fruits were wrapped in newspaper and transported in thermal boxes to the Product Technology Laboratory of the Federal University of Pará, Campus Altamira-PA. In the laboratory the fruits were sanitized in chlorinated solution (5 $mg.L^{-1}$) for 1 minute and dried at room temperature (25 °C) for a period of six hours.

Packaging and treatment with potassium permanganate

After drying, the fruits were randomly distributed in styrofoam trays of polystyrene (3 und) covered with plastic film with 14 micron PVC plastic film with and without the presence of the sachets with potassium permanganate ($KMnO_4$) and stored in a cold room at the temperature of 13 °C for a total period of 20 days.

The sachet with potassium permanganate was prepared as described by Ferreira (2009). After preliminary tests evaluating concentrations ranging from 1 to 6 grams (g) of the product, the sachet containing 3 g of $KMnO_4$ was chosen based on the results presented. For this purpose, 3 g of pure $KMnO_4$ for analysis (PA) and 6.5 g of fine texture vermiculite were weighed. The potassium permanganate was dissolved in deionized water and the solution was placed in contact with the vermiculite previously autoclaved. The material was kept in an oven (80 °C) until complete drying. After drying, the vermiculite impregnated with $KMnO_4$ was placed in TNT (non-woven fabric) sachets and disposed inside the packages with the fruits.

Physicochemical analysis

The fruit quality analyzes were determined on the following variables:

Weight loss: Determined by weighing the fruits using a precision scale (0.1 g) by calculating the difference in mass on the initial day and that obtained for each evaluation period and the results were expressed as a percentage (%).

Firmness of the fruit: Determined by the penetration force, necessary for the needle of 2.5 cm in length and 0.8 cm in diameter perforate the bark of the fruit. A hand pressure penetrometer (Model FT 011) was used, and measurements were taken at 3 points of the median area of the fruits and expressed in Newton (N).

External Appearance: Determined using seven trained raters who assigned grades on a five-point hedonic scale as per Lima et al. (2004), where: 5 = absence of depressions, wilt or attack of microorganisms; 4 = traces of depressions and/or wilting; 3 = slight depressions and/or wilting; 2 = depressions and/or wilt with medium intensity and mild attack of microorganisms and 1 = depressions and wilt with severe intensity and attack of microorganisms. Note 3 was considered the limit for marketing.

Cracking index: Determined by visual analysis on the surface of the peel of the fruits the appearance of cracks and counting performed at each time of storage (Barbosa et al., 2011).

Starch content: 80 g of the pulp was homogenized with 100 ml of distilled water, from which 20 g of the mixture which was rested for 12 hours in a beaker containing 50 ml of 95% ethyl alcohol at 50 ° C was removed. After this time, the mixture was filtered on filter paper and the residue was used for starch analysis. The preparation of the extract was performed according to methodology described by Aguiar (2013). For quantification, 1.5 mL of the extract and 1 mL of the cupric reagent were used, which after vortexing were kept in a boiling water bath for 15 minutes. After cooling, the solution was added 1 mL of the arsenic-molybdic reagent, 3 mL of distilled water and then the spectrophotometer was read at 510 nm and the results were expressed as a percentage.

Soluble solids: Determined by refractometry using a 2g grinding of the fruit pulp and a digital refractometer of the brand Atago, model N-1α, with reading in the range of 0 to 95 ° Brix, and the results were expressed in ° Brix (AOAC, 2012).

Titratable acidity: Determined by the titration of 10 mL of homogenized juice through a Mix (brand name Walita 400 Watt) with 90 mL of distilled water. 0.2 N NaOH solution was used as the titrant by adding three drops of 1% phenolphthalein as indicator to the sample. The results were expressed as eq.mg citric acid.100 mL^{-1}.

pH: determined using a digital potentiometer (model AK 90) calibrated with buffer solutions 4.0 and 7.0 using 10 g of homogenized fruit pulp with 50 mL of distilled water (AOAC, 2012).

Ratio (SS/AT): determined by the ratio of soluble solids to titratable acidity.

Color: The color analysis was performed at 3 points in the median region of each fruit by means of a Color Flex 45/0 (2200) colorimeter, stdzMode: 45/0 with direct reflectance reading of the L* coordinates (brightness) a* (red or green tint) and b* (yellow or blue tint) of the Hunterlab Universal Software system. From the values of L* and a* b*, the *hue* angle (° h*) and the chroma saturation index (C*) were determined. For each repetition, the average of four measurements per fruit was used.

Cold Damage: Determined by means of visual analysis with the aid of seven trained evaluators who assigned grades on a hedonic scale: 0 = absence of spots and spots on the bark; 1 = small brown spots; 2 = small brown spots; 3 = darker and larger spots. The result was expressed as a percentage (%).

Experimental design and Statistical analysis

The experimental design was completely randomized in a 2x5 factorial arrangement consisting of two treatments (control and with $KMnO_4$ sachets, 3 g) and five evaluation times (0, 5, 10, 15 and 20 days) with five replicates and the experimental plot composed of three fruits.

The data of each variable were submitted to analysis of variance (ANAVA) being analyzed statistically through the SAS (Release 4.5, StatSoft, Inc., USA) and the means compared by means of the Tukey test at the level of significance of 0.05%.

RESULTS AND DISCUSSION

The packaging of pine cones with potassium permanganate sachets (KMnO4) significantly reduced ($p < 0.05$) the loss of weight and the incidence of cracks, besides maintaining the firmer fruits and with better visual aspect when compared to the fruits of the control treatment (Fig. 1A, B, C and D), respectively.

The observed increase in fruit mass loss with storage time is a result of the loss of water and solutes through respiration, which after harvest is intense in climacteric fruits such as pinha. However, fruit packing with $KMnO_4$ sachets contributed to the lower mass loss (7.85%) compared to 11.15% of the control treatment after 20 days of storage (Fig. 1A). This lower loss in the treated fruits is possibly related to the decrease of the respiratory activity, due to the lower accumulation of ethylene inside the packages, agreeing with Silva et al. (2010) that in work with 'Golden' papaya observed loss of mass for the upper control treatment (5.5%) to treatments with $KMnO_4$ (3.7%) after 15 days of storage at 25° C.

In the case of anonaceae such as pinhas, the water loss is directly related to the external appearance, the softening of the pulp and, consequently, the incidence of cracking. In this sense, the delay of the physiological processes resulting from the action of ethylene through the use of sachets impregnated with $KMnO_4$ resulted, for example, in the lower index of cracks (9.56%) in relation to control fruits (13.5%) (Fig. 1B), on firmer fruits (26.3N) to 18.54 N of the control treatment (Fig. 1C) reflecting the best visual appearance with a score of 3.55 (mild depressions/wilting) compared to note 2.25 (depressions/wilting with medium intensity and mild attack of microorganisms), respectively (Fig. 1D) at the end of the storage period.

The delay in ripening also resulted in greater firmness in fruits of atemóia (Reis, 2013), banana 'Grand Nine' (Sarka et al., 2017) and nectarine (Jayarajan e Sharma, 2018), besides better visual appearance of mangabas (Nasser et al., 2015), when they were packed with $KMnO_4$ sachets.

The delay in the maturation of fruits packed with $KMnO_4$ sachets ensured less starch degradation (Fig. 2A) and in the synthesis of soluble solids (Fig. 2B) during the storage period ($p<0.05$) in relation to control fruits.

The storage time showed a reduction in starch content and an increase in soluble solids content (SS) in fruit pulp. The degradation of the starch is a characteristic feature during the ripening process of climacteric fruits, because as it is hydrolyzed by the respiratory metabolism, there is an increase in the total soluble sugars contents (Chitarra and Chitarra, 2005; Silva et al., 2009). Similarly, Mizobutisi et al. (2012) and Silva et al. (2013) also observed increased soluble solids contents in response to starch degradation in pinheira fruits stored at 12 °C and 15 °C for 18 and 21 days, respectively.

The most significant reduction in starch content occurred from the 10º day of storage in both treatments, however the use of $KMnO_4$ sachets by absorbing the exogenous ethylene inside the packages delayed the ripening and,

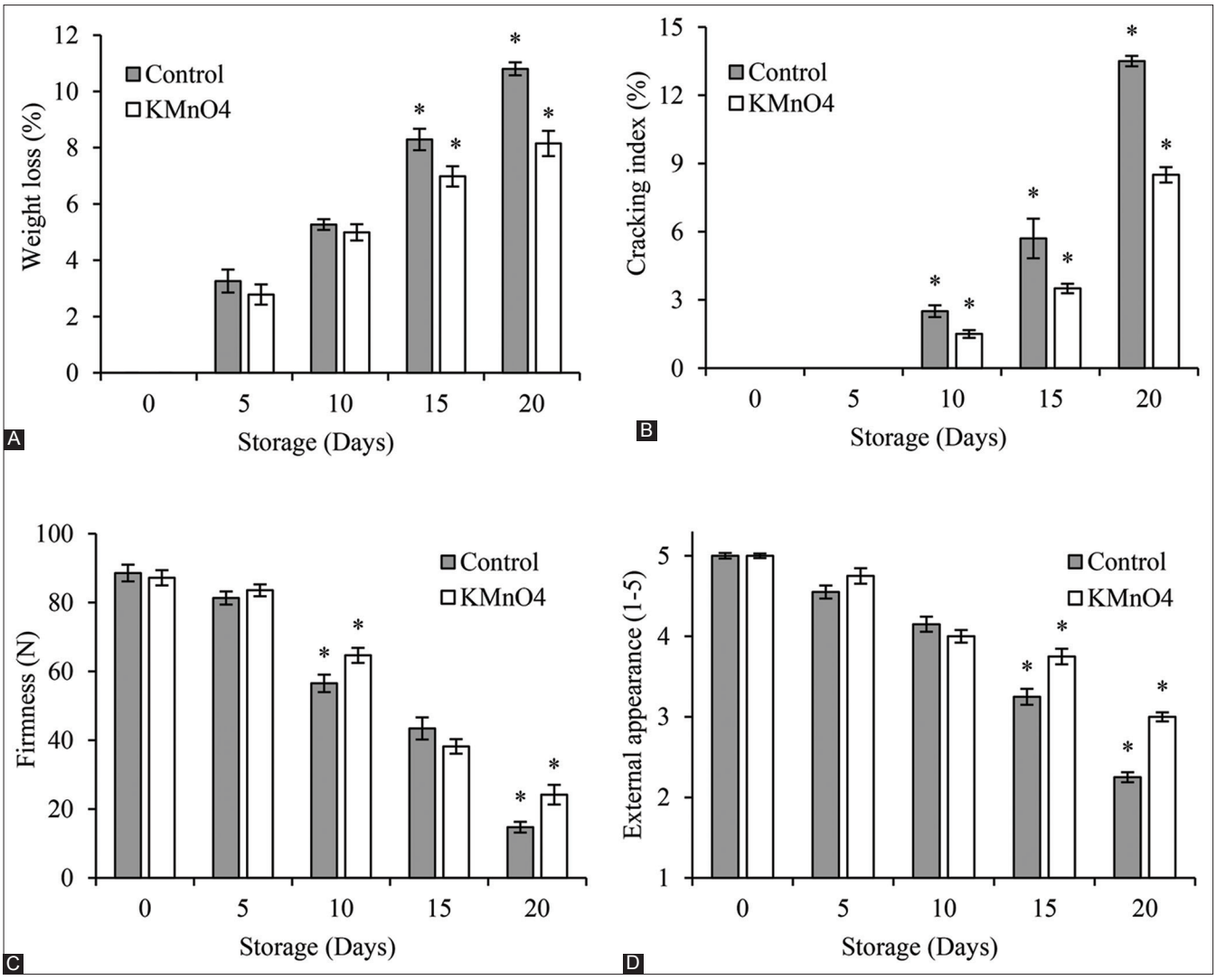

Fig 1. Weight loss (A), Cracking index (B), firmness (C) and external appearance (D) of pinhas packed with potassium permanganate sachets ($KMnO_4$, 3 g) and stored under refrigeration (13 °C) for 20 days. * Represents a significant difference between treatments within each storage time, $p < 0.05$.

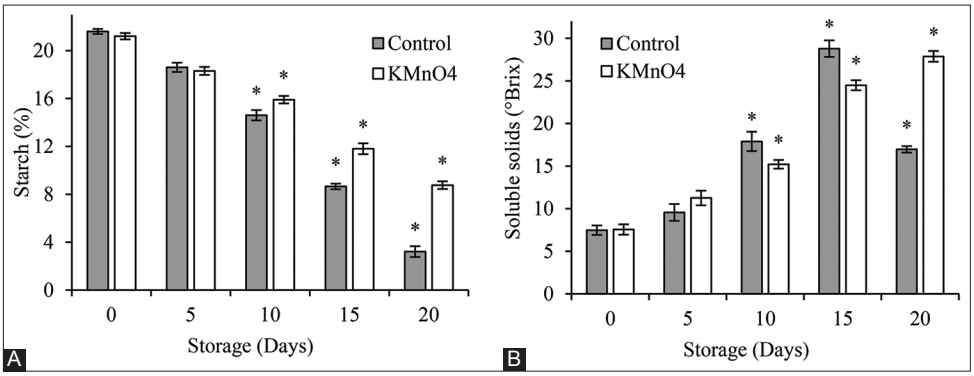

Fig 2. Starch content (A) and soluble solids contents (B) of pinhas packed with potassium permanganate sachets ($KMnO_4$, 3 g) and stored under refrigeration (13 °C) for 20 days. *It represents a significant difference between treatments within each storage time, $p<0.05$.

consequently, the conversion of the starch to soluble sugars was slower (8.15%) than the control fruits (3.87%) after 20 days of storage.

In this sense, the increase in soluble solids content (SS) during storage occurred in response to starch hydrolysis. The maximum SS values occurred at the 15º and 20º days

for the control fruits (28.78 ºBrix) and when packed with $KMnO_4$ sachets (27.56 ºBrix), respectively. The gradual increase in SS content in the fruits stored with the ethylene absorber demonstrates its efficiency in delaying maturation in relation to control fruits whose decline after 15 days suggests the use of soluble sugars as a source of energy in respiratory metabolism. In 'Sunrise Golden' papaya packed with $KMnO_4$ sachets and stored at 10 °C for 25 days a lower elevation in solids content of 2 g (SILVA et al., 2009). Sarkar et al. (2017) found that 'Grand Nine' bananas packed with sachets containing 5 g of $KMnO_4$ and maintained at 25 ºC showed the lowest variations in SS content at the end of storage (19º day), probably in response to lower fruit ripening speed.

The acidity of the fruits presented variations with storage time (Fig. 3A), while pH showed a tendency to decrease (Fig. 3B) showing significant differences ($p<0.05$) between treatments.

The fruit acidity showed an increase up to the 10º day in the control fruits (0.43 mg citric acid.$100g^{-1}$) and at the 15º day in the fruits packed with $KMnO_4$ sachets (0.47 mg citric acid.$100g^{-1}$), after These periods show a reduction in values until the end of the storage period. According Chitarra e Chitarra (2005) the decrease in the values of titratable acidity occurs due to the consumption of organic acids, in this case the citric acid, due to the respiratory process. Despite this decrease, higher titratable acidity values ($p<0.05$) (0.34 mg citric acid.$100g^{-1}$) were observed in fruits packed with $KMnO_4$ sachets in relation to the control fruits (0.27 mg acid citric acid.$100g^{-1}$) assuming a less advanced stage of ripening due to the slower consumption of organic acids.

In studies on the storage of pinhas (Silva et al., 2003; Vila et al., 2005; Mizobutsi et al., 2012) observed an increase in acidity with a subsequent reduction in the final third of fruit storage, associating this variation with the ripening of fruits through the consumption of acids in respiration. There was a reduction in pH values for both treatments during the storage period from 6.45 on day zero to 5.71 after 20 days. Some studies point to a clear trend between the reduction of pH and the advance of maturation of the anonaceae (Melo et al., 2002).

In summary, fruit packing with $KMnO_4$ sachets resulted in a slower pH decline exhibiting a significant difference ($p<0.05$) on days 5, 15 and 20 of the storage periods. This result is due to the absorption of the ethylene that surrounds the fruits, delaying the biochemical transformations responsible for the changes in the pH of the fruits. On the other hand, Nasser et al. (2015) in research with mangaba e Falcão et al. (2017) with bananas 'Prata Anã' and 'Grand Nine', did not observe influence of the $KMnO_4$ sachet on the pH and acidity of the fruits during 15 and 30 days of storage at 3 and 13 ºC respectively.

The SS/TA flavor ratio (Fig. 4A) was intensified with storage time and treatments ($p<0.05$). However, the color of the fruits on the variables *hue* (Fig. 4B), chroma (Fig. 4C) and luminosity (Fig. 4D) presented influence of the sachet of $KMnO_4$ ($p<0.05$) only for *hue* angle.

The SS/AT ratio is one of the most widely used forms of flavor evaluation and is more representative than the isolated measurement of sugars or acid, since it gives a good idea of the balance between these two components (Chitarra and Chitarra, 2005). In this study, the increase in the SS/TA ratio over 20 days reached an average value of 95.5, corroborating that found by Silva et al. (2013) which obtained a mean value of SS/TA (97.1) in pinhas after 21 days of storage at 15 ºC. On the other hand, Reges et al. (2018) found values greater than 190 after 8 days of storage at 25 °C. This variation in the relation and taste (SS/TA) is influenced by both environmental and physiological factors that interfere in the metabolism of sugars and acids and, consequently, the fruit's flavor. Considering that the increase in SS/TA ratio is a clear indicative of sweetness, the lower ratio (90.76) observed in

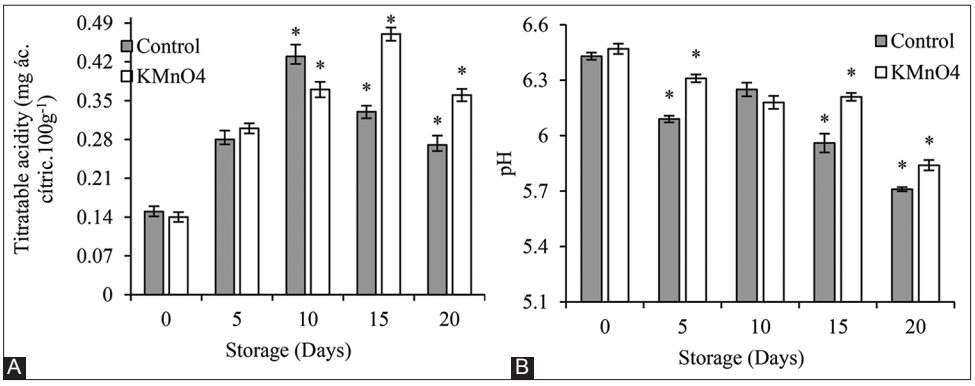

Fig 3. Titratable acidity (A) and pH (B) of pinhas packed with potassium permanganate sachets ($KMnO_4$, 3 g) and stored under refrigeration (13 °C) for 20 days. *It represents a significant difference between treatments within each storage time, $p<0.05$.

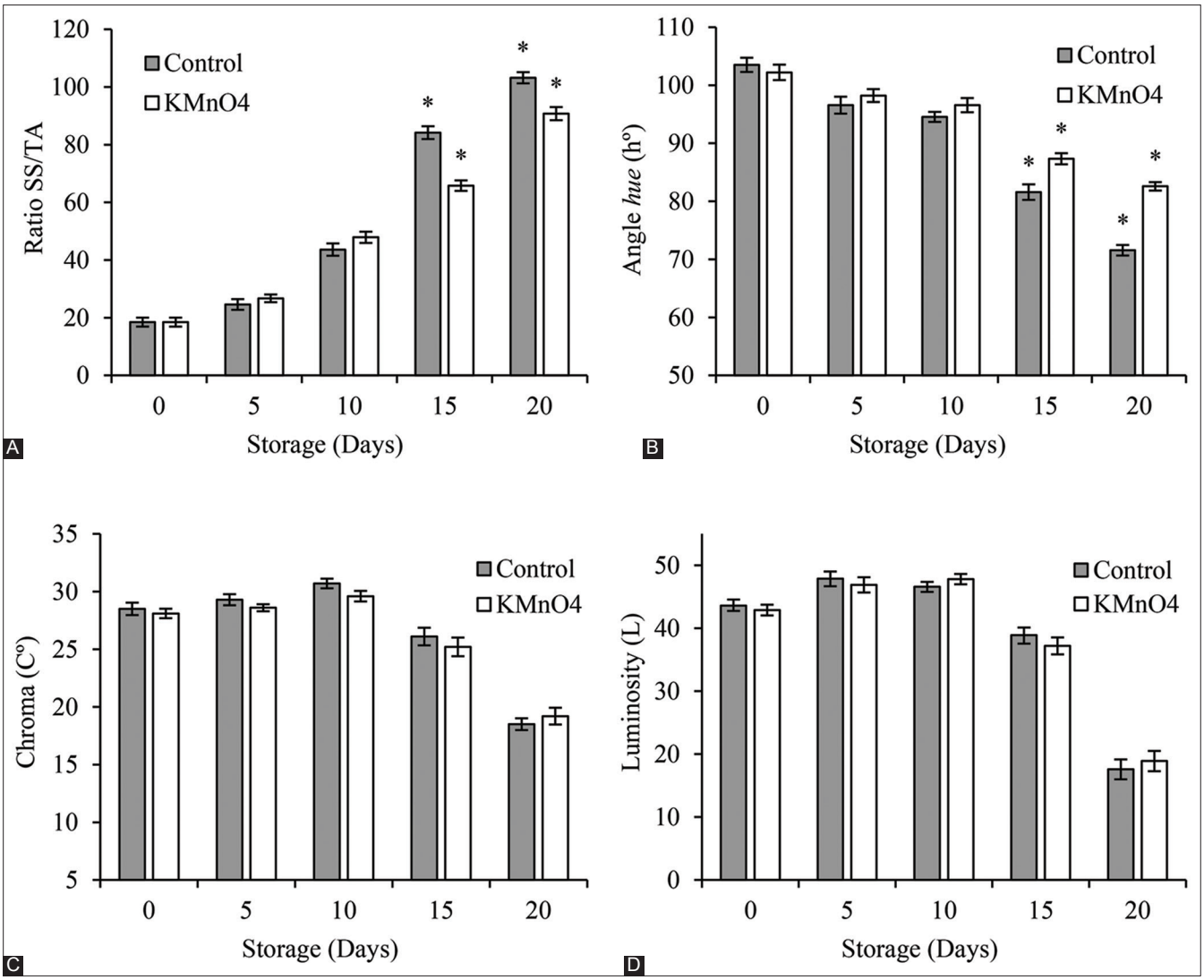

Fig 4. Ratio SS/TA (A), angle hue (B), chroma (C) and luminosity (D) of pinhas packed with potassium permanganate sachets ($KMnO_4$, 3 g) and stored under refrigeration (13 °C) for 20 days. *It represents a significant difference between treatments within each storage time, $p<0.05$.

fruits packed with $KMnO_4$ sachets suggests a less advanced stage of maturation ($p < 0.05$) to control fruits (103.19) at the end of storage.

Regarding the color of the fruits, there was a decrease in the values of ºhue with the storage time (Fig. 4B) indicating that the color of the peel changed from green to yellow, however, the removal of ethylene through the $KMnO_4$ sachets delayed the degradation of chlorophyll, that is, kept the fruits greener (82.59 h) differing ($p<0.05$) from the control fruits (71.56 h) after 20 days. Regarding the color of the fruits, there was a decrease in the values of angel *hue* with the storage time (Fig. 4B) indicating that the color of the peel changed from green to yellow, however, the removal of ethylene through the $KMnO_4$ sachets delayed the degradation of chlorophyll, that is, kept the fruits greener (82.59 h) differing ($p < 0.05$) from the control fruits (71.56 h) after 20 days. Corroborating with Moradinezhad and Jahani (2019) where' Shahroudi' apricot packaging with $KMnO_4$ sachets (2 and 5 g) delayed fruit color loss after four weeks of storage at 0.5 °C relative to their control.

For color intensity, chroma (Fig. 4C) and luminosity (Fig. 4D) no significant difference ($p>0.05$) was observed between the treatments. For both variables, there was a reduction in mean values, especially after the 10º day of storage, reaching average values of 18.6 and 17.9 for chroma and luminosity at the end of 20 days, respectively. It is possible that this decrease is associated with the beginning of browning of the bark due to the progress of ripening, chilling injury and senescence itself.

There were cases of cold damage during the storage of pinhas, but without significant effect ($p> 0.05$) of the sachet with $KMnO_4$ in relation to its control (Fig. 5).

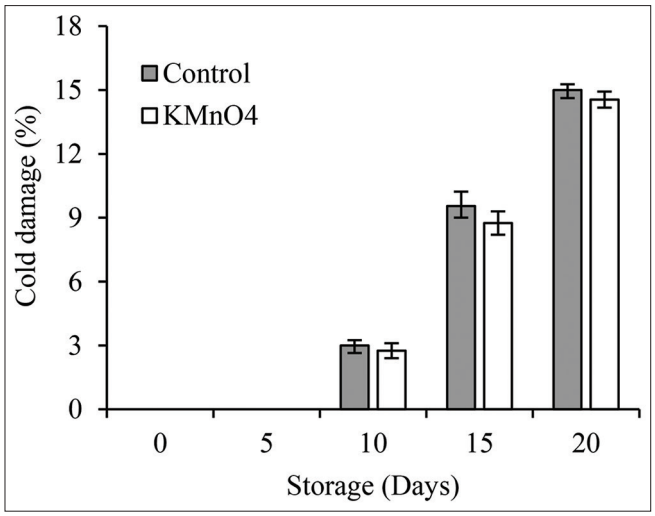

Fig 5. Cold damage of pinhas packed with potassium permanganate sachets ($KMnO_4$, 3 g) and stored under refrigeration (13 °C) for 20 days. *It represents a significant difference between treatments within each storage time, p<0.05.

The cold damage occurred on the 10º day of storage of fruits with 3.05% in the control fruits and 2.75% in those packaged with $KMnO_4$ sachets, so that at day 20 the percentages corresponded to 14.55 and 14.00, respectively. The cold damage represents the physiological dysfunction "*chilling injury*" that occurs as a function of the time of exposure of the fruits at low temperature, in the case of the pinhas e inferior to 13 ºC. In this sense, it is assumed that the occurrence of cold damage in pine cones is associated with other metabolic processes that are not linked to ethylene synthesis.

CONCLUSION

The packaging of pinhas with potassium permanganate sachets ($KMnO_4$, 3 g) delays maturation preserving the physicochemical characteristics during 20 days of storage at 13 ºC.

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