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# Drying and storage of macaúba fruit: chemical and oxidative stability

# Secagem e armazenamento de frutos de macaúba: estabilidade química e oxidativa

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**Highlights**:
Post-harvest of bocaiuva fruits (*Acrocomia* sp); drying and storage.
Physical analysis of fruit and pulp. Chemical analysis of pulp and almond;
Extraction and quality of almond pulp and oil.

## Abstract

The objective of this study was to evaluate the chemical and oxidative stability of dried macaúba (*Acrocomia totai*) fruit in different air conditions and stored for up to 120 days. The macaúba fruits were collected, sanitized, dried at the temperatures of 40, 50, 60 and 70 °C and stored in raffia bags at room temperature. Drying at higher temperatures resulted in pulps with a darker and orangish color and pulp oil with lower titratable acidity contents and acidity indices. Throughout storage, there was an increase in the acidity index of pulp oil and a reduction in the ascorbic acid and carotenoid contents in the pulp. Drying and storage reduced the ascorbic acid and carotenoid contents of the pulp. Drying at lower temperatures results in pulps with a higher free-radical sequestering ability. Pulp oil quality was compromised by drying and storage time. Newly harvested macaúba fruits can be dried at 40, 50, 60 or 70 °C and stored for up to 120 days without compromising nut oil quality.
**Key words**: *Acrocomia totai*. Antioxidant. Temperature. Dehydration. Oil.

## Resumo

Este trabalho teve por objetivo avaliar a estabilidade química e oxidativa de frutos de macaúba desidratados em diferentes condições e armazenados por até 120 dias. Os frutos de macaúba "*Acrocomia totai*" foram coletados, sanitizados, desidratados nas temperaturas de 40, 50, 60 e 70 °C e armazenados em sacos de ráfia em temperatura ambiente. A secagem em temperaturas mais elevadas resultou em polpas com coloração mais escura e alaranjada, porém com menor acidez titulável e índice de acidez do óleo da polpa. Durante o armazenamento houve aumento do índice de acidez do óleo da polpa e redução do teor de ácido ascórbico e carotenoides da polpa dos frutos. A secagem e o tempo de

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armazenamento dos frutos diminuíram o teor de ácido ascórbico e de carotenoides da polpa. A secagem em temperaturas mais baixas resultou em polpas com maior capacidade de sequestrar radicais livres. A qualidade do óleo da polpa foi comprometida pela secagem e tempo de armazenamento. Frutos de macaúbas recém coletados podem ser desidratados a 40, 50, 60 ou 70 °C e armazenados por até 120 dias, sem comprometer a qualidade do óleo da amêndoa.
**Palavras-chave:** *Acrocomia totai.* Antioxidante. Temperatura. Desidratação. Óleo.

## Introduction

Bocaiuva, also known as macaúba, is the fruit of a native palm tree of the genus *Acrocomia*, found in all regions of Brazil, in the phytogeographic biomes of Amazon, Caatinga, Cerrado, Atlantic Forest and Pantanal (Leitman, Soares, Henderson, Noblick, & Martins, 2018). Two of the six species are the most largely used by local populations in the country: *A. aculeata* and *A. totai* (Silva, 2017).

Macaúba has a high lipid content 30.38-44.20% in the pulp and 41.10-55.55% in the nut and nutritional properties that enable its use as food. In the pulp, these fats consist mostly of monounsaturated fatty acids, especially oleic acid (omega 9), whereas in the nut, saturated fatty acids predominate, with lauric acid occurring in the highest percentage (Munhoz, Guimarães, Nozaki, Sanjinez-Argandoña, & Macedo, 2018). The fruit contains β-carotene and α-tocopherol, which are natural antioxidants and precursors of vitamins A and E, respectively (Coimbra & Jorge, 2011). These substances are related to reduced risk of cardiovascular disease and non-transmissible chronic diseases like cancer (Fisk II, 2011).

The use of macaúba fruits, especially in the Brazilian state of Mato Grosso do Sul, has a strong socioeconomic and environmental appeal, as it is an abundant natural resource with the potential to be used for various purposes, mainly as a food. It is of extreme importance for the conservation of biodiversity and income generation for extractive communities and family farmers (Silva, Campos, Borsato, Cândido, & Donadon, 2018). The macaúba fruit can be consumed fresh or processed as a meal (Kopper, Saravia, Ribani, & Lorenzi, 2009) obtained by manual or mechanical extraction (I. A. Amaral, Loubet Filho, Cavalheiro, Galvani, & Santos, 2019). According to P. B. Silva et al. (2019), the mechanically extracted edible oil from its pulp has antidiabetic and antioxidant properties, which enable its inclusion in functional foods.

Due to the commercial potential of macaúba as raw material for the pharmaceutical, cosmetic, food and biofuel industries (Chuba-Machado, 2018) and the impossibility of immediately processing the fruit harvested from the season crop, drying is an essential step for storage, since, as described by Ball (2013), it allows the removal of part of the water contained in agricultural products. In this way, field losses are minimized and the product can be stored longer, preventing the development of microorganisms.

Increased temperatures reduce the drying time of macaúba fruit (Gonçalves, 2018), speeding up this process during the season harvest and reducing the subsequent waiting time, thereby preventing deterioration. However, Gonçalves (2018) stated that increases in the drying temperature affected oxidative stability and the acidity index of oils extracted from the macaúba mesocarp for biofuel production. The authors then recommended a drying temperature of up to 56.6 ºC

On these bases, the present study examined the chemical and oxidative stability of macaúba fruit dried in different air conditions and stored for up to 120 days.

## Material and Methods

Macaúba branches were harvested manually after physiological development, in the municipality of Corumbá - MS, Brazil, in December 2016. Injured,

green or senescing fruits were discarded. Whole fruits with a predominant yellow color in the peel were sanitized with chlorinated water (200 mg $L^{-1}$) for 10 min. Water on the fruit surface was dried on a countertop lined with paper towel. The fruits were divided into two batches: Batch 1, containing approximately 3 kg of fruit, was used for the physicochemical characterization, whereas Batch 2, with 100 kg, was used for drying in different air conditions.

The fresh fruit was characterized physically in terms of equatorial and longitudinal diameters, total weight, water content and peel and pulp color, by separating 25 units for the assessments. External diameter was measured using a digital caliper (ZAAS 6), with results expressed in millimeters. The fruit, peel (epicarp), pulp (mesocarp) and seed (nut) weights were determined using an analytical balance (AND HR-202), with results expressed in grams. The fruit yields as peel, pulp and nut were determined by subtracting the peel, pulp and nut weights from the total weight, respectively, and correlating each fraction to the total mass; results were expressed in percentage terms.

The water content of whole fresh fruits was determined in accordance with V. M. Silva et al. (2017), while the water activity of the fresh pulp was measured using a water activity meter (Hydropalm Aw1).

Fresh peel and pulp color was determined using a spectrophotometer (CM-2300d, Konica Minolta), with results expressed as L*, a* and b*.

To determine the maturation stage of fresh fruits, as proposed by the Instituto Adolfo Lutz (2008), the pulps were evaluated for soluble solids content (SS), titratable acidity (TA; method 312 IV), SS/TA ratio and pH, in three replicates.

The fresh-fruit pulp was characterized regarding the lipid contents (method 032 IV) and the pulp and nut oils were evaluated for the acidity, peroxide and iodine indices, methods 325 IV, 326 IV and 329 IV of the Instituto Adolfo Lutz (2008). To determine these indices, the lipids of both fractions (pulp and nut) were cold-extracted by immersing the sample, inside a filter-paper cartridge, in petroleum ether (boiling point 30-40°C) for 24 h, without heating, and separating the solvent in a rotary evaporator (892 d, Fisatom, Brazil).

The ascorbic acid content of the fresh pulp was determined by method 365 IV of IAL (2008), whereas the carotenoid content was measured as proposed by Rodriguez-Amaya (2001). The antioxidant activity of the pulp was measured by method DPPH (2,2-diphenyl-1-picrylhydrazyl), in an adaptation suggested by Fukumoto and Mazza (2000), using hydrocetonic extracts (70% acetone), according to Roesler et al. (2007). Analyses were carried out on microplates by serially diluting the sample. The results for ascorbic acid, carotenoids and IC 50 were corrected for samples with a 12% wet basis (wb) moisture content.

The macaúba fruits were artificially dried in a forced-air oven (Marconi, MA-035/5) at the temperatures of 40, 50, 60 and 70 ºC. The decrease in water content throughout the drying process was monitored by the gravimetric method, which required knowledge of the initial water content of the product, until the desired water content was reached (<15% wb).

Drying-air temperature was monitored using a thermometer that was installed in the dryer, and air relative air humidity was calculated based on the basic principles of psychrometry, by monitoring the internal (temperature) and external (temperature and relative humidity) air conditions of the dryer. The obtained values were then used to determine air relative humidity in the dryer, using Grapsi_draw 4.0 software. The average temperature and relative humidity values in the dryer were 41.33±1.54 °C and 31.72±2.99%; 52.2±2.39 °C and 18.96±2.08%; 59.1±0.74 °C and 14.21±2.07%; and 70±0.1 °C and 8.50±0.66% when they were regulated at 40, 50, 60 and 70 ºC, respectively. Fruit drying time at 40 ºC was 91 h; at 50 and 60 ºC, 42 h; and at 70 ºC, 27 h (Figure 1).

After drying, the fruits were packed in raffia bags and stored at room temperature for up to 120 days. Temperature and relative humidity during the storage period were monitored using a thermohygrometer.

The temperature in the storage room ranged between 25-30 ºC and relative humidity was 61%. The minimum air relative humidity dropped from 54 to 40%, whereas the maximum humidity did not vary considerably, ranging between 57 and 62%.

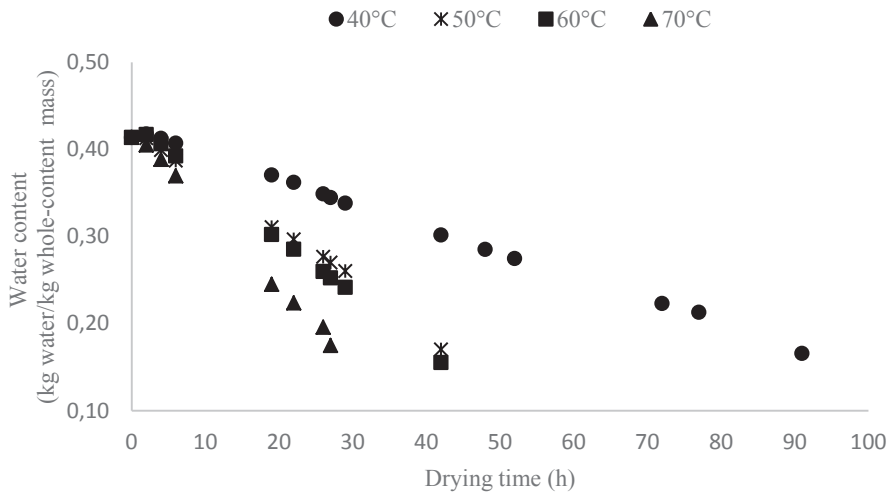


**Figure 1.** Drying curve of macaúba fruits dried at different temperatures.

During storage, the pulps of the fruits from each treatment were chemically evaluated at 0, 30, 60, 90 and 120 days (five periods) in three replicates, using 350 g of product per replicate. The pulp was evaluated for the water content, by the oven-drying method, until reaching a constant weight (IAL, 2008); water activity (Hydropalm Aw 1); color, by spectrophotometry (Konica Minolta CM-2300d); lipid content (method 032 IV); titratable acidity, using alcohol as a solvent (to facilitate sample dilution and prevent the formation of lumps) (method 415 IV); ascorbic acid, by method 365 IV (IAL, 2008); total carotenoids (Rodriguez-Amaya, 2001); and antioxidant activity (Roesler et al., 2007). Values were expressed as 12% (wb) moisture.

The pulp and nut oils were evaluated for the acidity, peroxide and iodine indices, following methods 325 IV, 326 IV and 329 IV, respectively, of the IAL (2008). Lipids were cold-extracted using petroleum ether as solvent (30-40 ºC) and separated in a rotary evaporator (Fisatom 802d, Brazil) (IAL, 2008).

The drying-storage experiment was set up as a completely randomized design with a 4 × 5 factorial arrangement represented by four drying temperatures (40, 50, 60 and 70 °C) and five storage times (0, 30, 60, 90 and 120 days), in three replicates. Each replicate was evaluated in triplicate. Data were subjected to analysis of variance and means were compared by Tukey's test at the 1% probability level. When the Treatment × Storage time interactions were significant, the interaction effects were decomposed with the factors analyzed within each level of another factor.

## Results and Discussion

The macaúba fruits showed an individual weight of 29.83±1.48 g and equatorial and longitudinal diameters of 32.94±2.79 and 32.18±2.27 mm.

Similar values were found in macaúba fruits harvested in Dourados-MS (33.39 and 34.68 mm, respectively) and Presidente Epitácio - SP (31.65 and 33.14 mm, respectively), with no significant differences occurring between both locations (Sanjinez-Argandoña & Chuba, 2011). Higher diameter values were reported in macaúba fruits in Belo Horizonte - MG, 44.70±2.2 and 43.60±1.74 mm, respectively) (Queiroz, 2016). The great variability found in those physical traits is possibly related to genetic diversity and soil-climatic factors.

The whole fruits showed 41.36±3.23% moisture and the pulp showed a water activity of 0.94±0.01%. Likewise, the water activity in macaúba pulp found by Sanjinez-Argandoña and Chuba (2011) was also high (0.90 to 0.95). Storage resistance is associated with the metabolic activity of tissues, which decreases as the water content in the plant tissue is reduced (Chitarra & Chitarra, 2005). Water activity is characterized by the available water content, which is related to enzymatic activity and microbial proliferation. According to Ferreira Neto, Figueirêdo and Queiroz (2005), most microbiological agents grow in medium with water activity in the range of 0.90 to 0.99.

The peel and pulp of the fresh fruits showed positive a* (peel = 12.73±3.81; pulp = 14.29±1.02) and b* (peel = 35.87±3.58; pulp = 46.61±5.85) values, which represent a red-to-yellow tone, respectively. The higher b* values in comparison to a* indicate a predominant yellow color. However, the pulp shoed elevated lightness values (L*= 67.74±0.67), resulting in a lighter yellow tone than the peel (L*= 46.61±5.85).

Fresh macaúba pulp showed SS and TA values of 12.80 °Brix and 0.18±0.00 g 100 $g^{-1}$ citric acid, respectively. The SS/TA ratio was 71.11, which indicates the stage of maturation, considering the SS content and the low TA content; and also a sweet pulp, which can thus be used in the making of ice creams and jams. The pH value (5.74±0.02) suggests a pulp of low acidity, similar to the pulp of fruits harvested in the Pantanal region of MS State (Souza, Cuellar, Donadon, & Guimarães, 2019). These results confirmed the adequate time of harvest adopted in this study.

The lipid content of fresh macaúba pulp was 15.47±1.01 g 100 $g^{-1}$, which is similar to the 13.96-15.77% found by Silva et al. (2018) in the pulp of macaúba fruits obtained in the state of Mato Grosso do Sul (13.96-15.77%) and lower than the 44.2±0.2% found by Queiroz (2016) in macaúba fruits in Rio Largo - AL.

The acidity, peroxide and iodine values of fresh macaúba pulp oil were 5.31 mg NaOH $g^{-1}$, 3.73 mEq $kg^{-1}$ and 56.90 g $I_2$ 100 $g^{-1}$, respectively, whereas the respective nut oil indices were 0.54 mg NaOH $g^{-1}$, 0.00 mEq $kg^{-1}$ and 18.36 g $I_2$ 100 $g^{-1}$. These acidity and peroxide values are within the accepted range for palm oil (<10 mg KOH $g^{-1}$) and virgin olive oil (<20 mEq $kg^{-1}$), respectively, and the iodine index of the pulp oil is lower than the 73.25±0.10 obtained by Oliveira (2016) in macaúba pulp oil in São Paulo. The iodine index increases proportionally to the level of unsaturation; accordingly, a higher level of unsaturated fatty acids was found in the pulp compared to the nut. Pulp oil consists mostly of long-chain fatty acids (F. P. Amaral, Broetto, Batistella, & Jorge, 2011).

Fresh macaúba pulp showed an ascorbic acid content of 14.54±0.00 mg 100 $g^{-1}$, which is similar to the 11.46 mg.100 $g^{-1}$ found in macaúba pulp harvested in Presidente Epitácio - SP, but conflicting with the 34.57 mg.100 $g^{-1}$ observed in macaúba fruit in Dourado - MS (Sanjinez-Argandoña & Chuba, 2011). The total carotenoid content of the pulp was 77.69±0.34 μg $g^{-1}$, which is close to the value obtained by Souza, Cueller, Donadon and Guimarães (2019) in the pulp of macaúba fruit harvested in the Pantanal region of MS.

Microbial activity in the fresh pulp was high ($IC_{50}$ = 0.49±0.00 mg $mL^{-1}$), considering that lower values indicate greater antioxidant capacity (Luzia & Jorge, 2010).

The water content of the whole fruit and the water activity in the pulp of macaúba fruits dried in different air conditions (40, 50, 60 and 70 ºC) did not differ between the temperature groups (P<0.01), with moisture values between 11.24 and 12.01%. There was no statistical difference for these values during the storage period (30, 60, 90 and 120 days) (Table 1). Therefore, the variations in air relative humidity during storage did not interfere with the water content of the fruits during the evaluated periods. The observed water content values were lower than the maximum limit (15%) for meals (RDC n° 263 of September 22, 2005), whereas the Aw values were lower than the minimum limit (0.60) for the development of microorganisms in dried products (Pinto & Neves, 2010).

**Table 1**
**Water content of the fruit, Aw, color (L*, a*, b*) and titratable acidity of macaúba pulp dried in different air conditions and stored for up to 120 days**

| Treatment | Moisture (%)** | Aw | L* | a* | b* | Acidity** (g 100 $g^{-1}$) |
|---|---|---|---|---|---|---|
| 40 ºC | 12.01 a | 0.36 a | 64.87 a | 9.73 b | 32.11 a | 0.36 b |
| 50 ºC | 11.90 a | 0.38 a | 64.93 a | 9.62 b | 28.83 b | 0.42 a |
| 60 ºC | 11.24 a | 0.37 a | 64.45 a | 9.91 b | 27.64 b | 0.33 c |
| 70 ºC | 11.27 a | 0.33 a | 59.54 b | 11.66 a | 25.07 b | 0.23 d |
| F test | 1.30 NS | 2.79 NS | 142.50** | 75.53 ** | 153.43** | 339.11** |
| Time (days) | | | | | | |
| 0 | 13.18 a | 0.35 a | 61.95 b | 11.11 a | 30.67 a | 0.24 d |
| 30 | 11.02 a | 0.36 a | 64.48 a | 9.99 b | 29.27 a | 0.30 c |
| 60 | 10.89 a | 0.35 a | 64.47 a | 9.84 c | 27.29 b | 0.36 b |
| 90 | 10.83 a | 0.35 a | 63.79 b | 9.96 b | 27.86 b | 0.36 b |
| 120 | 12.12 a | 0.39 a | 62.55 b | 10.27 b | 26.97 b | 0.41 a |
| F test | 6.60 NS | 1.28 NS | 21.90 ** | 40.18 ** | 35.12 ** | 176.03 ** |
| Treatment × Time | 0.95 NS | 1.26 NS | 7.10 ** | 32.09** | 11.58** | 25.82 ** |
| CV (%) | 0.06 | 0.05 | 0.03 | 0.03 | 0.08 | 0.19 |

Means followed by the same lowercase letter in the columns do not differ from each other according to Tukey's test (P<0.01).
**Calculation, 12% moisture base.

In the analysis of pulp color parameters, significant differences were found for the interaction between drying temperatures and storage times. The fruits which had been dried at 70 ºC showed significant differences for pulp color, with the lowest L* value, indicating a darker tone than the others, caused by the increase in drying temperature. The lightness of the pulp of macaúba fruits dried at 60 and 70 ºC did not change during storage (Table 2). When the storage time of macaúba dried at 40 and 50 ºC was evaluated, lighter pulps were observed when the fruits were dried at 40 ºC and stored for 60 days and at 50 ºC and stored for up to 30 days (Table 2).

Drying at 70 ºC resulted in pulps with a redder tone compared to those of fruits dried at lower temperatures (Table 1). During storage, the a* values of the pulps of the fruits dried at 40 ºC decreased, indicating a reduction of red intensity. This trend was also noted for the pulps of the fruits dried at 50 °C and stored for up to 90 days. At 120

days of storage, the a* values of the pulp of fruits dried at 50 ºC did not differ from those found in the pulps of fruits newly dried (0 days) at the same temperature. The pulp a* values did not change throughout storage in the fruits dried at 60 and 70 ºC (Table 2).

The b* values were higher in the pulps from the fruits dried at 40 ºC and stored for up to 30 days (Table 1). Higher b* values mean yellower pulps. The decomposition of the significant interactions revealed that the b* values in the pulp decreased during the storage of fruits dried at 40 and 50 ºC, indicating a reduction in the yellow tone. The pulps of fruits dried at 60 and 70 ºC showed no significant changes in color during storage (Table 2).

**Table 2**
**Changes in color (L*, a* and b*) and soluble titratable acidity of macaúba pulp dried in different air conditions and stored for up to 120 days**

| Treatment | Storage time (days) | | | | |
|---|---|---|---|---|---|
| | 0 | 30 | 60 | 90 | 120 |
| | | | Lightness | | |
| 40 °C | 62.32 Ba | 65.10 Ba | 67.82 Aa | 65.42 Ba | 63.70 Ba |
| 50 °C | 62.15 Ba | 67.49 Aa | 65.46 Ba | 66.06 Ba | 63.47 Ba |
| 60 °C | 63.92 Aa | 64.64 Aa | 65.79 Aa | 64.81 Aa | 63.11 Aa |
| 70 °C | 59.40 Aa | 60.70 Ab | 58.81 Ab | 58.85 Ab | 59.92 Ab |
| | | | a* | | |
| 40 °C | 12.42 Aa | 9.66 Bb | 8.58 Bc | 9.22 Bb | 8.79 Bc |
| 50 °C | 10.54 Ab | 8.50 Bc | 9.25 Bb | 8.97 Bb | 10.84 Ab |
| 60 °C | 9.66 Ac | 10.25 Ab | 9.71 Aa | 9.78 Ab | 10.15 Ab |
| 70 °C | 11.81 Aa | 11.53 Aa | 11.80 Aa | 11.88 Aa | 11.29 Aa |
| | | | b* | | |
| 40 °C | 38.25 Aa | 33.06 Ba | 29.54 Ca | 31.00 Ca | 28.69 Ca |
| 50 °C | 31.43 Ab | 29.03 Ba | 27.73 Ba | 28.86 Ba | 27.09 Ba |
| 60 °C | 28.64 Ab | 28.09 Aa | 27.54 Aa | 26.48 Aa | 27.43 Aa |
| 70 °C | 24.35 Ab | 26.88 Aa | 24.34 Aa | 25.11 Aa | 24.68 Aa |
| | | | Soluble titratable acidity (g citric acid 100 $g^{-1}$) | | |
| 40 °C | 0.21 Cb | 0.31 Bb | 0.42 Aa | 0.40 Aa | 0.46 Ab |
| 50 °C | 0.28 Db | 0.40 Da | 0.42 Ca | 0.44 Ba | 0.57 Aa |
| 60 °C | 0.28 Ba | 0.28 Bb | 0.36 Aa | 0.35 Aa | 0.35 Ac |
| 70 °C | 0.21 Ab | 0.21 Ac | 0.25 Ab | 0.25 Ab | 0.25 Ad |

Means followed by the same lowercase letter in the column and uppercase letter in the row do not differ from each other according to Tukey's test (P<0.01).

By comparing the results of pulp color in the dried compared to the fresh fruit (L= 61.74±0.67, a*= 14.29±1.02 and b*= 46.61±5.85), we observe that the former showed lower a* and b* values (Table 1). When the values were projected onto the color diagram (hue and saturation), the dried pulps were found to have a more orangish tone as temperature and storage time increased.

The pulp of fruits dried at 60 and 70 ºC showed lower titratable acidity (Table 1), indicating that

the lower rate of decrease in water content at the temperatures of 40 and 50 ºC and the longer exposure to heat until the expected water content was reached favored the degradation of lipids from the pulp and the release of free acids. In the fruits dried at 70 ºC, pulp acidity remained stable throughout storage, whereas in the fruits dried at 40, 50 and 60 ºC, the acidity contents increased, indicating degradation of the existing lipids (Table 2).

The results presented in Tables 3 and 4 reveal that higher drying temperatures and longer storage times resulted in decreased ascorbic acid and carotenoid contents. Accordingly, the pulps had a higher free-radical sequestering ability when the fruits were dried at 40-50 ºC and stored for up to 90 days. The decomposition of the significant interactions showed that the ascorbic acid contents did not change in the fruits dried at 70 ºC, during storage (Table 3). Results for the ascorbic acid content were close to those found by Reis, Figueiredo, Ferraz and Freitas (2017) in seedless acerola (*Malpighia emarginata*) meal.

**Table 3**
**Bioactive compounds in macaúba pulp dried in different air conditions and stored for up to 120 days (20-30 ºC; 40-61% RH)**

| Treatment | Ascorbic acid* (mg 100 $g^{-1}$) | Carotenoids* (μg $g^{-1}$) | Antioxidant activity* $IC_{50}$ (mg $mL^{-1}$) |
|---|---|---|---|
| 40 ºC | 5.45a | 36.10 a | 6.59 b |
| 50 ºC | 3.85b | 29.14 b | 7.02 b |
| 60 ºC | 3.95b | 23.38 d | 9.18 a |
| 70 ºC | 3.91b | 25.86 c | 7.74 a |
| F test | 86.59** | | |
| Time (days) | | | |
| 0 | 5.24a | 41.45 a | 6.49 b |
| 30 | 4.90a | 28.74 b | 6.61 b |
| 60 | 4.33b | 26.70 c | 6.33 b |
| 90 | 3.43b | 25.46 c | 8.63 a |
| 120 | 3.56c | 20.74 d | 10.10 a |
| F test | 72.93** | 406.89** | 11.13 ** |
| Treatment × time | 21.15** | 118.29** | 3.84** |
| CV (%) | 0.21 | 0.23 | 0.18 |

Means followed by the same lowercase letter in the columns do not differ from each other according to Tukey's test at the 1% probability level.
*Calculation, 12% moisture base.

Results for ascorbic acid and carotenoids in the fresh pulp (14.54±0.00 mg 100$g^{-1}$ and 77.69±0.37 μg $g^{-1}$, respectively) were higher than those found after drying, reinforcing the fact that the drying process reduces those contents. In a study on macaúba jam, the total carotenoid contents found in the macaúba pulp and jam with passion fruit were 58.29 and 11.44 mg 100 $g^{-1}$, respectively, demonstrating the reduction post-processing. In the same study, the ascorbic acid content detected in macaúba pulp was minimal, with the different morphotypes found in the state of Mato Grosso do Sul providing different pulp color tones and compositions (Souza, Cueller, Donadon & Guimarães, 2019).

The total carotenoid content was statistically higher in the pulp of the fruits that were dried at 40 and 50 ºC. Similarly, Chuba-Machado (2018) reported that pulp of macaúba fruits dried at 50 ºC showed higher carotenoid values (64.6 to 78.3 μg $g^{-1}$) than samples of fruits dried at 70 ºC (56.9 to 62.6 μg $g^{-1}$).

The decreasing carotenoid contents may explain the results obtained for color, where the dried fruits showed lower a* and b* values compared to the fresh fruits. This was possibly due to the degradation of those pigments, which confer tones that range from red to yellow (Chitarra & Chitarra, 2005).

In this way, the less intense color corresponded to a lower carotenoid content. All treatments exhibited a reduction in total carotenoid content throughout the storage period. According to Rios, Antunes and Bianchi (2009), throughout the storage period, carotenoids may be isomerized and oxidized due to their unstable structure, and the increase in temperature and exposure to light are related to oxidation or decomposition of the carotenoid chain.

Compared to the results for $IC_{50}$ obtained with the fresh pulp (0.49±0.00 mg $mL^{-1}$), fruit drying was shown to reduce free-radical sequestering ability. The lower $IC_{50}$ values in the pulp of fruits dried at 40-50 °C indicated greater free-radical sequestering ability, which was not observed in the fruits dried at 60-70 ºC. During storage, the values remained stable for up to 90 days at the temperatures of 40 and 60 ºC and up to 60 days at 70 ºC (Table 3). At 90-120 days of storage, higher values were seen (Table 4), suggesting a lower free-radical sequestering ability.

**Table 4**
**Change in ascorbic acid and carotenoid contents and antioxidant activity of pulp of macaúba fruit dried in different air conditions and stored for up to 120 days (20-30 ºC; 40-61% RH)**

| Treatment | Storage time (days) | | | | |
|---|---|---|---|---|---|
| | 0 | 30 | 60 | 90 | 120 |
| | Ascorbic acid* (mg 100 $g^{-1}$) | | | | |
| 40 °C | 7.53 Aa | 7.13 Aa | 5.16 Ba | 3.74 Ca | 3.71 Ca |
| 50 °C | 4.54 Ab | 4.41 Ab | 4.37 Aa | 2.94 Ba | 3.01 Ba |
| 60 °C | 5.20 Ab | 4.43 Ab | 3.37 Bb | 3.32 Ba | 3.42 Ba |
| 70 °C | 3.67 Ab | 3.64 Ab | 4.40 Aa | 3.71 Aa | 4.11 Aa |
| | Carotenoids* (μg $g^{-1}$) | | | | |
| 40 °C | 54.71 Aa | 48.19 Aa | 35.89 Aa | 23.28 Bb | 18.43 Bb |
| 50 °C | 49.95 Aa | 16.32 Dc | 30.30 Bb | 25.97 Cb | 23.13 Ca |
| 60 °C | 30.93 Ab | 25.40 Bb | 18.57 Bc | 21.33 Bb | 20.66 Ba |
| 70 °C | 30.20 Ab | 25.04 Bb | 22.05 Bc | 31.27 Aa | 20.73 Ba |
| | Antioxidant activity* $IC_{50}$ (mg $mL^{-1}$) | | | | |
| 40 °C | 4.97 Ba | 6.89 Ba | 5.83 Ba | 4.44 Bb | 10.80 Aa |
| 50 °C | 7.48 Aa | 6.18 Aa | 4.79 Aa | 10.16 Aa | 6.47 Aa |
| 60 °C | 8.05 Ba | 7.77 Ba | 9.01 Ba | 8.72 Bb | 12.37 Aa |
| 70 °C | 5.45 Ba | 5.61 Ba | 5.68 Ba | 11.20 Aa | 10.74 Aa |

Means followed by the same lowercase letter in the columns and uppercase letter in the row do not differ from each other according to Tukey's test ($P<0.01$).
*Calculation, 12% moisture base.

Ferreira, Santos, Costa and Gebara (2013) developed a shake-type beverage prepared from dry extract of macaúba pulp, obtained by freeze-drying. The beverage could be considered an innovative functional food, as it can reduce the risk of diseases, especially those related to excess free radicals.

The pulps from fruits dried at the different temperatures did not differ from each other for the lipid content (17.66±0.03 g 100 $g^{-1}$, 12% wb of moisture). The oil content in the macaúba mesocarp was not influenced by drying-air temperature, either (Gonçalves, 2018).

In the pulp oil, drying the fruit at 70 ºC resulted in the lowest acidity index (Table 5), which differed statistically from the results obtained at the other drying temperatures. The obtained values are within the threshold established by the Brazilian Health Surveillance Agency (Anvisa) (10 mg KOH $g^{-1}$) (RDC n° 270 of September 22, 2005) for virgin palm oil.

**Table 5**
**Acidity, peroxide and iodine indices of pulp and nut oil from macaúba fruits dried in different air conditions and stored for up to 120 days**

| Treatment | OIL (PULP) | | | OIL (NUT) | | |
|---|---|---|---|---|---|---|
| | Acidity index (mg NaOH $g^{-1}$) | Peroxide index (mEq $kg^{-1}$) | Iodine index (g $I_2$ 100 $g^{-1}$) | Acidity index (mg NaOH $g^{-1}$) | Peroxide index (mEq $kg^{-1}$) | Iodine index (g $I_2$ 100 $g^{-1}$) |
| 40 ºC | 12.62 b | 16.83 b | 60.32 a | 0.14 a | 0.00 | 26.04 a |
| 50 °C | 16.74 a | 22.90 b | 54.28 a | 0.15 a | 0.00 | 22.88 a |
| 60 °C | 13.17 b | 33.28 a | 59.53 a | 0.13 a | 0.00 | 20.56 b |
| 70 °C | 3.04 c | 35.95 a | 41.91 b | 0.14 a | 0.00 | 21.48 a |
| F test | 268.14 ** | 88.61 ** | 13.90 ** | 1.24 ** | 0.00 | 5.26 ** |
| Time (days) | | | | | | |
| 0 | 6.94 e | 11.63 d | 60.29 a | 0.31 a | 0.00 | 19.84 b |
| 30 | 9.77 d | 26.09 c | 53.36 a | 0.11 b | 0.00 | 24.53 a |
| 60 | 13.00 b | 39.08 a | 56.03 a | 0.11 b | 0.00 | 27.93 a |
| 90 | 11.66 c | 32.43 b | 42.50 b | 0.08 b | 0.00 | 24.21 a |
| 120 | 15.60 a | 26.98 c | 55.87 a | 0.10 b | 0.00 | 17.18 b |
| F-test | 67.66 ** | 147.23 ** | 8.04 ** | 86.89 ** | 0.00 | 12.38 ** |
| Treatment × time | 10.75 ** | 26.81 ** | 7.22 ** | 3.01 ** | 0.00 | 4.19 ** |
| CV (%) | 0.37 | 0.29 | 0.13 | 0.36 | 0.00 | 0.14 |

Means followed by the same lowercase letter in the columns do not differ from each other according to Tukey's test (P<0.01).

Oil acidity remained elevated at the other drying temperatures (40, 50 and 60 °C) throughout the storage period (Tables 3 and 4), except for 70 ºC, in which case it remained low. According to Pohndorf (2012), increased oil acidity indices result from the hydrolytic rancidity process, which might or might not have occurred by an enzymatic action, considering that the decomposition of glycerides is accelerated with exposure to light. Thus, the temperature of 70 ºC inhibited alterations in the pulp due to the shorter fruit storage time until the desired moisture was achieved. Increasing acidity indices in the pulp oil were also reported by Queiroz (2016) during the storage of macaúba fruit for up to 30 days, after they were subjected to different harvest/cleaning methods, followed by oven-drying at 60 ºC for 24 h (or no drying).

Results pertaining to the peroxide index in the pulp oil from the dried fruits (Table 5) indicated that exposure to heat significantly influenced the degree of oxidization, which was highest at the temperatures of 60 and 70 ºC. Overall, storage time contributed to the increase in peroxide index (Tables 5 and 6). The fruit pulps provided an oil that met the standards of quality for this index as established by Anvisa (20 mEq $kg^{-1}$) (RDC n° 270 of September 22, 2005) for virgin palm oil when the material is dried at the temperatures of 40 to 50 ºC (0 days). This suggests that, after drying, the storage conditions did not prevent oxidization, which was potentiated by the increase in drying temperature.

**Table 6.**
**Chances in acidity, peroxide and iodine indices of pulp and nut oil from macaúba fruits dried in different air conditions and stored for up to 120 days**

| Treatment | Storage time (days) | | | | |
|---|---|---|---|---|---|
| | 0 | 30 | 60 | 90 | 120 |
| Acidity index of pulp oil (mg NaOH $g^{-1}$) | | | | | |
| 40 °C | 8.50 Ca | 11.87 Ba | 11.35 Bb | 12.86 Ba | 18.56 Ab |
| 50 °C | 8.71 Ca | 13.26 Ba | 21.71 Aa | 15.93 Ba | 24.09 Aa |
| 60 °C | 8.12 Ba | 11.84 Aa | 15.40 Ab | 14.92 Aa | 15.56 Ab |
| 70 °C | 2.44 Ab | 2.10 Ab | 3.53 Ac | 2.95 Ab | 4.20 Ac |
| Peroxide index of pulp oil (mEq $kg^{-1}$) | | | | | |
| 40 °C | 4.94 Bb | 20.49 Ab | 20.16 Ab | 23.32 Ab | 15.25 Ab |
| 50 °C | 5.32 Cb | 25.97 Ba | 41.91 Aa | 21.46 Bb | 19.85 Bb |
| 60 °C | 17.44 Ca | 24.26 Ba | 51.16 Aa | 43.56 Aa | 29.99 Bb |
| 70 °C | 18.80 Aa | 33.65 Aa | 43.10 Aa | 41.37 Aa | 42.81 Aa |
| Iodine index of pulp oil | | | | | |
| 40 °C | 61.72 Aa | 66.42 Aa | 60.98 Aa | 60.30 Aa | 52.17 Aa |
| 50 °C | 63.19 Aa | 62.96 Aa | 57.29 Aa | 35.96 Aa | 51.98 Aa |
| 60 °C | 59.70 Aa | 69.33 Aa | 72.28 Aa | 33.76 Aa | 62.59 Aa |
| 70 °C | 64.57 Aa | 14.71 Bb | 33.55 Ba | 39.96 Aa | 56.75 Aa |
| Acidity index of nut oil (mg NaOH $g^{-1}$) | | | | | |
| 40 °C | 0.35 Aa | 0.09 Ba | 0.14 Ba | 0.07 Ba | 0.07 Ba |
| 50 °C | 0.26 Aa | 0.17 Ba | 0.09 Ba | 0.08 Ba | 0.16 Ba |
| 60 °C | 0.31 Aa | 0.08 Ba | 0.08 Ba | 0.08 Ba | 0.08 Ba |
| 70 °C | 0.33 Aa | 0.08 Ba | 0.14 Ba | 0.08 Ba | 0.08 Ba |
| Iodine index of nut oil (g $I_2$ 100 $g^{-1}$) | | | | | |
| 40 °C | 23.05 Aa | 27.73 Aa | 29.35 Aa | 26.48 Aa | 23.58 Aa |
| 50 °C | 12.18 Aa | 21.00 Aa | 36.12 Aa | 22.76 Aa | 22.36 Aa |
| 60 °C | 21.20 Aa | 25.00 Aa | 23.90 Aa | 21.03 Aa | 11.65 Aa |
| 70 °C | 22.94 Aa | 24.39 Aa | 22.36 Aa | 26.58 Aa | 11.14 Aa |

Means followed by the same lowercase letter in the columns and uppercase letter in the row do not differ from each other according to Tukey's test ($P<0.01$).

Exposure to heat interfered with the degradation reactions, considering that the fresh pulp, which was not dried artificially, exhibited lower acidity and peroxide indices in its oil (5.31 mg NaOH $g^{-1}$ and 3.73 mEq $kg^{-1}$, respectively).

The dried macaúba pulp oil showed a high iodine index, indicating a high number of unsaturated double bonds. The pulp oil from the fruit dried at 70 ºC showed a lower iodine index compared to those from fruits dried at the other temperatures, which did not differ from each other. Over the storage period, this index was lowest at 90 days of storage (Tables 5 and 6). The obtained iodine values were close to the 49.93 g $I_2$ 100 $g^{-1}$ found in guariroba oil (*Syagrus oleracea*) by Nozaki et al. (2012).

No significant difference was detected between the fruits dried at different temperatures for the lipid content in the nut (37.59±0.92 g 100 $g^{-1}$).

The nut oil acidity indices (Tables 4 and 5) differed statistically after the fruits were dried at different temperatures. Oliveira, Neves and Silva (2013) reported acidity values higher than those obtained in this study, in crude babassu (*Attalea speciosa*) oil (2.22 to 5.91 mg NaOH $g^{-1}$).

The peroxide index was not detected in the macaúba nut oil after the fruits were dried or during the storage period F. A. Silva et al. (2011) found peroxide indices of 2.32 to 2.57 mEq $kg^{-1}$ in macadamia nut.

Therefore, the acidity and peroxide indices in the nut were lower than the minimum of (<4 mg KOH $g^{-1}$ and <15 mEq $kg^{-1}$, respectively) established for crude oils (RDC n° 270 of September 22, 2005) until the end of the storage period.

Dried-macaúba nut oil showed a lower iodine index than the pulp oil, indicating a lower number of unsaturated double bonds. The obtained values were higher than the 7.34 g $I_2$ 100 $g^{-1}$ found in macaúba nut oil by Nozaki et al. (2012).

These results demonstrate that, irrespective of temperature and storage time, drying did not significantly influence the quality of macaúba nut oil possibly due to the predominance of saturated fatty acids in the nut and protection against heat transference provided by the endocarp to the seed.

## Conclusions

Drying and storage time reduce the ascorbic acid and carotenoid contents of fruit pulp. Lower temperatures provide pulps with a higher free-radical sequestering ability. Pulp oil quality was compromised by drying and storage. Newly harvested macaúba fruits can be dried at 40, 50, 60 or 70 °C and stored for up to 120 days without compromising nut oil quality.

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