DOI: https://doi.org/10.5327/fst.528



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## Evaluation of the freezing kinetics of araçá-boi pulp formulated with different maltodextrin concentrations

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#### **Abstract**

This study aimed to evaluate the freezing kinetics of araçá-boi pulp at  $-30^{\circ}$ C with different concentrations of maltodextrin. Formulations containing different concentrations (0, 7, 14, and 21%) of dextrose equivalent 10 maltodextrins were prepared. The pulps were characterized based on the following parameters: soluble solids, pH, titratable acidity, soluble solids/titratable acidity ratio, vitamin C, flavonoids, diffusion coefficient, model coefficient, effective diffusivity, average effective diffusivity, coefficient of determination, standard error of estimate, and mean relative error. For the freezing kinetics, a hole was made in the geometric center of the samples, and a thermocouple was inserted to monitor the internal temperature, while another thermocouple was placed inside the freezing unit to monitor the freezing medium, determining the thermal equilibrium point at  $-30^{\circ}$ C. The Fourier model applied to the experimental data showed coefficients of determination above 98% and low standard error of estimate and mean relative error values. The pH increased with higher maltodextrin concentrations, ranging from 2.46 to 2.55. Vitamin C content decreased as maltodextrin concentration increased, with the 14 and 21% formulations showing the lowest vitamin C values. The freezing curve was similar to that observed for pure water, allowing for the identification of the three stages involved in the water-to-ice phase transition. The freezing time increased proportionally with the addition of maltodextrin, with the longest times observed for the 21% maltodextrin formulation and the shortest for the control (0% maltodextrin) at  $-30^{\circ}$ C.

**Keywords:** *Eugenia stipitata*; myrtaceae; freezing curve; thermal stability.

**Practical Application:** Varying maltodextrin concentrations influence water activity, texture, and freezing rates. This helps tailor formulations based on desired shelf life, texture, or nutrient retention.

#### 1 INTRODUCTION

In Brazil, it is possible to find a wide variety of native fruits that can be used in the processing and development of new products. Among these, the Myrtaceae family stands out, with fruits from the Eugenia stipitata species, known as araçá-boi. This Amazonian fruit tree produces fleshy, globular fruits approximately 12 cm in diameter, with a mass ranging from 30 to 80 g (Borsoi et al., 2025). With a yellow coloration, the pulp can yield more than 80% when compared to the peel (Araújo et al., 2021), which is an excellent indicator for fresh market commercialization or further processing.

In addition to their physical properties, the fruits have flavors that make them sensorially attractive due to the presence of acids and their carbohydrate content. Characterized as an acidic fruit, araçá-boi stands out for its high levels of malic and cinnamic acids, as well as carbohydrates (65–72% w/w) and proteins (8–10% w/w) (Farias et al., 2023). These fruits, in addition to having a high nutritional composition, are rich in bioactive components classified as phenolic compounds, which exhibit high antioxidant capacity, helping to prevent pathological processes caused by oxidative stress in the body (Araújo et al., 2021; Gulcin, 2020).

Received: Jul. 11, 2025.

Accepted: Jul. 21, 2025.

Conflict of interest: nothing to declare.

Funding: nothing to declare.

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Biologically, fruits reach the senescence stage more rapidly after harvest during commercialization. Therefore, their high perishability requires the use of preservation methods that reduce both quantitative and qualitative losses, which would otherwise compromise their nutritional and sensory properties (Reyes-Alvarez & Lanari, 2023). Freezing is one of the simplest techniques used for fruit preservation, as it reduces physiological, biochemical, and microbiological activities, ensuring greater stability over a longer period (Zhao et al., 2024).

The cooling rate is one of the most important factors when using freezing as a method of food preservation, since it determines the size of the ice crystals formed during the process, elements directly related to the sensory quality of the products (Sutariya & Sunkesula, 2021). In some cases, the use of polymers can help improve or maintain the texture of pulps during the freezing stage, such as the use of maltodextrin (MD).

MD is a low dextrose equivalent (DE) agent that can encapsulate compounds by forming a film more rapidly and increasing the glass transition temperature ( $T_g$ ) of the mixture, thereby improving the quality and stability of pulps (Siccama et al., 2021). Considering the physicochemical and nutritional properties of araçá-boi and the importance of pulp stability in relation to freezing and MD concentrations, the present study aimed to evaluate the freezing kinetics of araçá-boi pulp at a temperature of  $-30^{\circ}$ C with different concentrations of MD.

#### 1.1 Relevance of the work

The study of the freezing kinetics of araçá-boi pulp with different concentrations of maltodextrin is highly relevant to food engineering. It provides key insights into thermal diffusion and physicochemical characteristics that directly affect the quality and stability of frozen products. The use of the Fourier model and analysis of parameters such as pH, vitamin C, and freezing time help optimize industrial processes, preserve nutrients, and enhance texture. Additionally, it contributes to the development of formulations with functional additives and promotes the value of native fruits within the food production chain.

### 2 MATERIAL AND METHODS

Araçá-boi fruits (*Eugenia stipitata*) were obtained from Fazenda Amizade, Vila Brasil – Una/BA. The samples were collected, stored in thermal containers, and transported to the Food Drying and Processing Laboratory at the Federal University of Campina Grande (UFCG), Campina Grande-PB. The fruits were selected, washed under running water, and sanitized in a sodium hypochlorite solution at 50 ppm for 15 min, followed by rinsing with potable water. Subsequently, the fruits were pulped using a horizontal pulper with a 0.8 mm diameter sieve. The pulp was homogenized and packaged in low-density polyethylene bags with a volume of 200 mL, then frozen and stored in a cold chamber at –18°C until the experiment was conducted. Analyses were performed at the Laboratory of Storage and Processing of Agricultural Products, in the Academic Unit of Agricultural Engineering at UFCG, Paraíba, Brazil.

#### 2.1 Formulations

Prior to the freezing stage and microstructure evaluation, it was necessary to prepare formulations containing different concentrations of DE 10 MD (0.7, 14, and 21%). The formulations were homogenized in a food processor (model 2, Britânia) for 60 s. The formulations were then distributed into polyethylene bags containing 100 g of sample, sealed, and the length, width, and height of each sample to be frozen were measured in millimeters (mm).

# 2.2 Physical and physicochemical characterization of the formulations

Water activity values were determined using an Aqualab device, and color was measured with a HunterLab colorimeter. Color values were expressed according to the CIELAB coordinate system, where the variables  $L^*$  (lightness),  $a^*$  (red-green component), and  $b^*$  (yellow-blue component) were used to calculate chromatic hue  $(h^\circ)$  and color saturation  $(C^*)$ .

Parameters analyzed included soluble solids content (SS in °Brix), pH, titratable acidity (TA), and the SS/TA ratio, according to Instituto Adolfo Lutz (IAL, 2008).

The determination of ascorbic acid content in the fruit pulps was based on the oxidation of ascorbic acid by the 2,6-dichlorophenolindophenol reagent (Association of Official Analytical Chemists [AOAC], 2007). Results were expressed as mg of ascorbic acid per 100 g of pulp.

Yellow flavonoids in the pulps were evaluated according to the methodology described by Francis (1982), based on extraction with ethanol (95%) and 1.5 M HCl (85:15, v:v). Absorbance readings were performed using a spectrophotometer, and yellow flavonoid content was expressed per 100 g of pulp as described by Silva et al. (2014).

#### 2.3 Microstructure evaluation

The microstructure of the pulp was analyzed using an optical microscope (HIROX KH1300) coupled with 2D Measure SOFTWARE, available at the Biomaterials Certification Laboratory (CERTBIO) of the Academic Unit of Materials Engineering at UFCG. Images of the structures were magnified at 200  $\times$  and 400  $\times$  and photomicrographed to observe the pulp structures after the irradiation process.

#### 2.4 Freezing kinetics

For the freezing kinetics experiment, a hole was made at the geometric center of each sample, and a thermocouple was inserted to monitor the temperature inside the sample. Another thermocouple was placed inside the freezing unit (horizontal freezer) to monitor the freezing environment, aiming to determine the time of thermal equilibrium at a temperature of  $-30^{\circ}$ C.

Using temperature values as a function of time (seconds), a graphical plot of the data was created using OriginPro 8 software. The freezing rate was determined according to Equation 1.

$$Vc = \frac{Ti - Tf}{\Delta \tau} (^{\circ}C. s^{-1})$$
 (1)  $A_n = \frac{2. sen \sigma_n}{\sigma_n + sen\sigma_n . cos\sigma_n}$  (4)

where  $T_i$  –initial temperature (°C);  $T_f$  –final temperature (°C);  $\Delta \tau$  – time required for the temperature to drop from  $T_i$  to  $T_f$ (s).

$$F_o = \left(\frac{\alpha}{L^2}\right).t\tag{5}$$

According to Cremers (1981), to calculate heat transfer under transient conditions in a way that resembles a plate with thickness 2L, for Fo =  $\alpha$  t L<sup>-2</sup>, the analytical solution can be expressed by Equations 2, 3, and 4 as follows:

$$RT = \sum_{n=1}^{\infty} A_n Exp(\sigma_n^2 F_o)$$
 (2)

Where

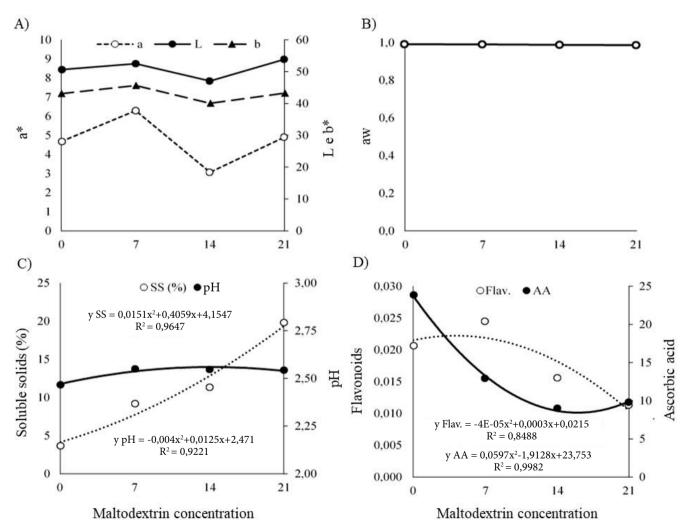
$$RT = \frac{T - T_{\infty}}{T_0 - T_{\infty}} \tag{3}$$

where RT = Temperature ratio, dimensionless; T = Temperature at each time, °C;  $T_{\infty}$  = Freezing medium temperature, °C;  $T_{\infty}$  = Initial temperature of the product, °C;  $F_{\infty}$  = Fourier number, dimensionless;  $A_n$  = Constant depending on the product;  $\sigma n$  = Transcendental root; L = Sample thickness divided by 2; t = Time, seconds;  $\alpha$  = Effective thermal diffusivity,  $mm^2 s^{-1}$ .

Using the temperature ratio data as a function of time, a nonlinear regression analysis was performed using Statistic software, version 7.0, to obtain the coefficients of Equation 5.

#### **3 RESULTS**

Figure 1 presents the values of physical, physicochemical, and bioactive compound characterization of whole and



**Figure 1**. Physical, physicochemical, and bioactive compound characterization of whole pulps and pulps formulated with different concentrations of maltodextrin. a\* – color intensity of red, b\* – color intensity of yellow, and L – luminosity (**A**); soluble solids and pH (**C**); and flavonoids and ascorbic acid (**D**).

formulated araçá-boi pulps. The L\* parameter is a measurement of lightness, evaluated on a grayscale ranging from black (0) to white (100) (Pathare et al., 2013). Regarding lightness, the L\* values varied with increasing MD concentration, ranging from 49.98 to 53.63. Canuto et al. (2010), when evaluating the characteristics of pulps from nine Amazonian fruit species, found a lightness value for araçá-boi pulp of  $40.7 \pm 0.3$ .

The addition of MD to the whole pulp reduced the moisture content due to the increased solids in the formulation, resulting in a significant difference. Total SS showed significant variations with the increase in MD concentration, with the highest average observed in the 21% formulation. The values obtained showed an average of 11.00 between the control sample and the MD concentrations.

The measured pH increased with the rise in MD concentration, ranging from 2.46 to 2.55. This average falls within the parameters found by Iturri et al. (2021), who analyzed araçá-boi pulp subjected to spray-drying and reported pH values of 2.71.

Vitamin C showed a significant reduction with the increase in MD concentration, where the 14 and 21% formulations resulted in lower vitamin C content values. The average vitamin C content was 13.91 mg of ascorbic acid per 100 g across all treatments. It was observed that there was no significant loss in flavonoid values; however, a decrease occurred with the increase in MD concentration when compared to the control treatment.

Figure 2 shows the freezing curves of the whole and formulated pulps (control, 0.7, 14, and 21% MD frozen at -30°C). The freezing curve of the whole pulp at -30°C (control – Figure 2) was very similar to the curve obtained for pure water, clearly allowing the distinction of the three typical stages during the conversion of water into ice. In the first stage, the cooling of the pulps is observed, where the temperature rapidly dropped from 23.3 to 2.3°C (whole pulp), 23.3 to 0.6°C (7% pulp), 24.5 to 1°C (14% pulp), and 21.9 to 0°C (21% pulp), taking on average 63 min for the duration of phase I.

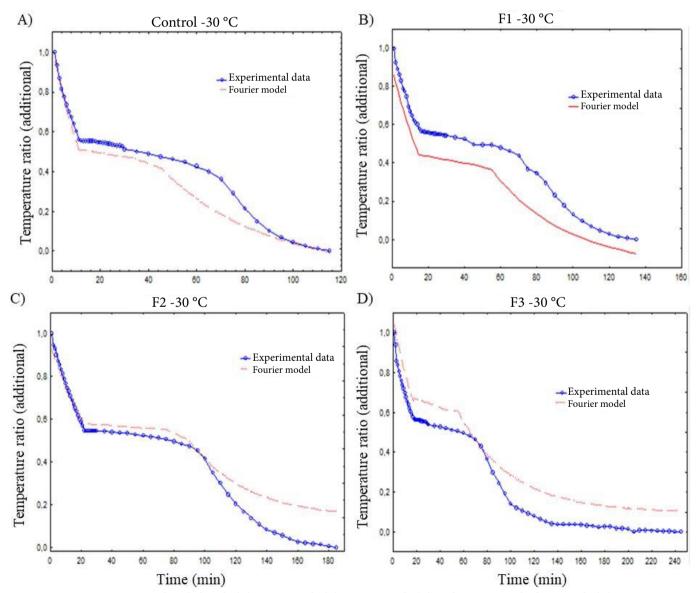
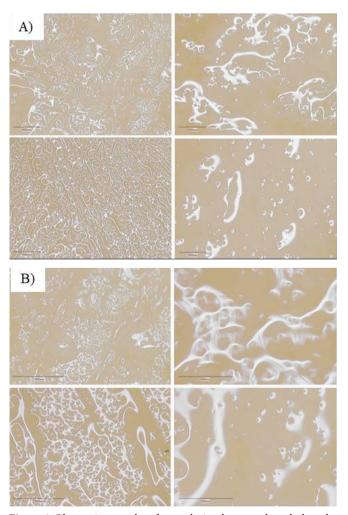


Figure 2. Freezing curve: control = whole pulp (A); F1 - 7% pulp (B); F2 - 14% pulp (C); and F3 - 21% maltodextrin pulp (D).

The freezing onset temperature was determined at the beginning of Phase II (nucleation). In the second stage, the phase change occurred slowly, with the temperature dropping from -0.1 to  $-2.6^{\circ}$ C (whole pulp), -0.1 to  $-2.4^{\circ}$ C (7% pulp), -0.2 to  $-2.5^{\circ}$ C (14% pulp), and -0.5 to  $-2.7^{\circ}$ C (21% pulp). The time required in the second stage was 30, 40, 75, and 40 min, respectively. The temperature ranged from -3.2 to  $-30.2^{\circ}$ C (whole pulp) in 115 min; -3.9 to  $-30.7^{\circ}$ C (7% pulp) in 135 min; -3 to  $-30.2^{\circ}$ C (14% pulp) in 185 min; and -3 to  $-30^{\circ}$ C (21% pulp) in 245 min.

Figure 3 shows the microstructures of the ice crystals of the free water in the araçá-boi pulp at  $-30\pm2^{\circ}\text{C}$ , observed at  $100\times$  and  $200\times$  magnifications. After freezing, the ice crystals exhibited branching structures similar to those of a tree, characterized as dendritic, which is present in most solidification processes.

Table 1 shows the parameters of diffusion coefficient, model coefficient (K), effective diffusivity ( $\alpha$ ), average effective diffusivity, coefficient of determination (R<sup>2</sup>), standard error of estimate (SE), and relative mean error (P%) for araçá-boi pulp during freezing stages at -30°C. It is observed that thermal diffusivity increases with MD concentration. The R<sup>2</sup> results were above 98%



**Figure 3**. Photomicrographs of araça-boi pulp control = whole pulp; F1 - 7% pulp; F2 - 14% pulp; F3 - 21% maltodextrin pulp at magnifications of  $100 \times (A)$  and  $200 \times (B)$ .

for all formulations, indicating a good fit of the data and the correct application of the Fourier equation to describe kinetic behavior. The Fourier model applied to the experimental data showed good coefficients of determination, above 98%, with low SE and *P*%.

#### 4 DISCUSSION

The pulps showed significant variations in luminosity among themselves, indicating a trend toward a lighter araçáboi pulp. It is noteworthy that the pulps with added MD had the highest values for this parameter, which was expected since the addition of MD increases L\* values due to its whiteness and powdery nature. Regarding the intensity of a\*, positive values corresponded to the red color of the samples, while positive b\* values corresponded to the yellow color; both were significantly influenced by the addition of MD.

The water content was influenced by the carrier agent, as observed by Ferrari et al. (2012) when studying the behavior of whole and formulated black mulberry pulps with 25% MD, which showed the formation of particles with lower moisture and hygroscopicity. The total SS found were higher than the values reported by Garzón et al. (2012) and Iturri et al. (2021), who reported SS contents of 4.6 °Brix for araçá-boi pulp. Therefore, the additional SS content resulting from MD is inversely proportional to the water content present in the pulps. The pH is an indicator of the pulp behavior regarding its sensory parameters with the addition of MD and can interfere with the final acidity of the pulps, as these two parameters show opposite behavior.

Bioactive compounds may undergo variations in their concentrations during processing due to their sensitivity to vegetable treatments. The araçá-boi pulps with MD showed a reduction in vitamin C content; however, it was possible to maintain higher levels than those reported in other studies, such as the one by Neri-Numa et al. (2013), who obtained average values of 11.34 when evaluating the antioxidant, antiproliferative, and antimutagenic potential of araçá-boi fruit.

Regarding flavonoids, higher results were found by Llerena et al. (2020), who assessed the effect of modified atmosphere packaging on the antioxidant capacity of araçá-boi, naranjilla, and tree tomato fruits, reporting an average value of 600.72 mg catechin per 100 g dry weight (DW) for the total flavonoid content of araçá-boi fruit. Therefore, the use of MD combined with other storage factors could help maintain higher bioactive quality in araçá-boi pulp.

Pereira and Resende (2020) stated that the initial stage of freezing consists of lowering the temperature below the freezing point of water, without a phase change. This point, unlike pure water, is always below 0°C due to the presence of dissolved solids in the product and occurs before the onset of ice crystal formation (Carvalho et al., 2017). The freezing onset temperature of ideal mono- and bicomponent solutions can be described by Raoult's law, but the specificity of fruit pulps, especially with the addition of solids, requires specific investigation to study the freezing onset temperature for each product. The freezing onset temperature (Tic) for fruit pulp systems depends on the same factors described for ideal solutions, with the solid content

**Table 1**. Parameters and coefficients of the Fourier model in the freezing kinetics of araçá-boi pulp with different concentrations of dextrose equivalent 10 maltodextrin.

Temperature	Formulation	Phases	$\alpha$ ( $\mathbf{m}^2\mathbf{s}^{-1}$ )	α average (m²s⁻¹)	$\mathbb{R}^2$	SE	P (%)
-30°C	Control	I	219.35	83.60	99.330	0.0000	.0000
		II	-		-	-	-
		III	92.76		84.325	0.0011	.0179
	F1 (7%)	I	192.76	100.11	94.775	0.0002	.0004
		II	-		-	-	-
		III	118.04		85.178	0.0005	.0212
	F2 (14%)	I	164.85	111.36	99.839	0.0001	.0002
		II	-		-	-	-
		III	163.24		90.906	0.0017	.0661
	F3 (21%)	I	164.37	121.54	97.171	0.0001	.0005
		II	-		-	-	-
		III	134.91		94.795	0.0009	.0498

being fundamentally important. The higher the solid content, the lower the initial freezing point will be (Salazar et al., 2018).

The experimental values determined are consistent with those reported in the literature: strawberry pulp frozen at  $-20^{\circ}$ C showed a Tic =  $-0.7^{\circ}$ C (Fernandes et al., 2010) and mangaba freezing showed a Tic =  $-1.0^{\circ}$ C (Soares et al., 2012). Pereira et al. (2013), freezing acerola pulp in a cold chamber at  $-25^{\circ}$ C, obtained a freezing onset temperature of  $-1.1^{\circ}$ C. During the freezing period, an increase in crystallization time was observed with the addition of MD to the samples; in this phase, ice crystals grow, latent heat is removed, and the concentration of SS increases SS (Soares et al., 2012). Phase III corresponds to post-freezing, during which the temperature of the already-frozen product decreases (Duarte et al., 2020).

The addition of solids to the material alters the freezing point of free water, which makes freezing more difficult; therefore, the total freezing time of araçá-boi pulp increased with the addition of MD to the product. The use of MD combined with freezing can promote changes in the physicochemical properties and microstructure of the raw material, stabilizing proteins and starch and altering the water content, which results in changes to the structure and composition of the cell walls (Wang et al., 2025).

According to Sousa et al. (2016), the thermal diffusivity of a material is influenced by the amount of water, temperature, composition, and porosity. Since water content and temperature of a product can vary considerably during processing, the value of thermal diffusivity also varies. In this context, the inclusion of MD altered the freezing rate. The increase in the solids content caused by the addition of MD was responsible for this behavior.

For freezing, knowledge of the thermal diffusivity coefficient is fundamentally important, as it is a transport property necessary for modeling and transient heat transfer calculations, as well as essential for simulation during freezing inside a food product, making it highly valuable for the industry, since without this thermophysical property, it would not be possible to calculate heat transfer in substances (Assegehegn et al., 2019).

#### **5 CONCLUSIONS**

The addition of MD causes significant changes in all analyzed physicochemical parameters, demonstrating that its use can directly affect the stability and sensory aspects of araçáboi pulp. The freezing time increases proportionally with the inclusion of MD in the araçáboi pulp, with the longest times observed for F3 (21% MD) and the shortest for the control (0% MD) at a temperature of  $-30^{\circ}$ C. The average effective diffusivity of the araçáboi pulp is directly proportional to the increase in thermal gradient and MD content.

#### **ACKNOWLEDGMENTS**

The authors would like to thank the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) for the financial support.

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