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Formulation and evaluation of guapeva jam: nutritional properties, bioactive compounds, and volatile compounds during storage

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Abstract

The guapeva (*Pouteria cf. gardneriana* Radlk.) is a Brazilian fruit that belongs to the Sapotaceae family and is considered a source of natural antioxidants and phenolic compounds; Thus, the objective of this work was to investigate the use of guapeva in the form of jam stored at different temperatures (25 °C and 35 °C) and to evaluate the degradation kinetics regarding the physicochemical parameters, bioactive compounds, and volatile profile, during the storage period of 150 days, in low-density polypropylene packaging's. The stored jams were mainly influenced by the storage time variable, with significant reductions in titratable acidity, pH, color, vitamin C, carotenoids, $ABTS^{\bullet+}$, DPPH, and volatile profile. The temperatures studied were not able to significantly affect the shelf life determined for the jams; however, the best temperature for storage stability of the guapeva jams was 25 °C since the most significant degradation of the compounds occurs with the use of 35 °C. The correlation between time and temperature had no significant effect on carotenoid content.

Keywords: bioactive compounds; jams; nutritional composition; *Pouteria* cf. *guardneriana Radlk*.

Practical Application: Storage time and temperature influence physicochemical parameters, bioactive compounds, and volatile profile quality of guapeva jam.

1 Introduction

The Brazilian flora has a great diversity of fruits with great agricultural potential and is still little explored technologically (Aguiar et al., 2019). The consumption of fruit with nutritive and functional value can be encouraged by using postharvest technologies that increase the useful life of these fruits (Arruda et al., 2016). Among the processes used to add value to fruits, the elaboration of jams, juice, and sweets is considered one of the most accepted forms by consumers (Curi et al., 2016).

Therefore, jams are obtained by cooking fruits with added sugar, edible acids, pectin, and water to the proper Brix grade for gelation, altering the osmotic pressure and product shelf life (Garrido et al., 2015). According to Mesquita et al. (2017), foods such as jams, juices, and sweets, are usually conserved by physical and chemical barriers such as preservative use and heat treatment. Yet, these foods suffer from some changes during storage. The storage conditions (packaging, storage time, and temperature) determine the degradation kinetics of food components, such as darkening and syneresis, and changes in antioxidant capacity, water activity, and texture.

The guapeva (*Pouteria cf. gardneriana* Radlk) belongs to the Sapotaceae family. The fruits are small and fleshy, typically consumed fresh or in products such as juice, sweets, wines, and syrup (Silva et al., 2012). Its small fruits are consumed due to their attractive color and particular taste and are considered sources of natural antioxidants, phenolic compounds, and highly nutritious (Barbosa et al., 2016).

The guapeva has colors that vary from yellow to orange when ripe, is oval-shaped with fine forage outside, pulp whitish and sweet, can contain one to four seeds located inland the fruit (Siqueira et al., 2017; Malta et al., 2013). Consumption of this fruit may contribute to the fight against the development of chronic degenerative diseases, such as cancer and diabetes. Besides natural consumption, exist the possibility to be used as a functional or micro-encapsulated ingredient for use as a drug, as it does not demonstrate any toxicological effects when tested on animals (Malta et al., 2013). Due to the nutritional potential of exotic fruits, this study aimed to evaluate the use of guapeva (*Pouteria cf. gardneriana* Radlk.) in the processed form of jams, replacing commercial pectin with passion fruit albedo, assessing the influence of temperature and time variables of storage in physicochemical parameters, bioactive compounds, and volatile profile during 150 days of storage in low-density polypropylene packages.

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2 Materials and methods

2.1 Processing and storage of guapeva jams

The guapeva (*Pouteria cf. gardneriana* Radlk.) was harvested in the Cerrado of the city of Palmas, capital of the State of Tocantins, Brazil (Latitude -10º18'43" S, Longitude -48º33'81" W) between August and November of 2019, due to its seasonal nature, with its fruiting being predominant between the months with less rainfall. The fruits were collected according to their stage of maturation and physical integrity. After the harvesting process, the fruits were transported in boxes to the Kinetics and Process Modeling Laboratory at the Federal University of Tocantins. They were sanitized and disinfected in 4% sodium hypochlorite solution for 30 minutes (Santos et al., 2017).

The fruits were submitted to manual extraction of peel/juice, and the pulp was obtained with a blender. The pulp was stored in polyethylene plastic packages and frozen at -20 °C until the jam was prepared. The guapeva jam presented in the formulation the following components: guapeva pulp (40%), crystal sugar (60%), monohydrated citric acid (1%), and passion fruit albedo as a source of pectin (3%). Guapeva jams were processed in an open stainless-steel pan, initially with pulp, sugar, and albedo. Citric acid was incorporated into the jams at the end of the cooking process to avoid pectin degradation due to acidity and high temperature. After processing the jams, the samples will be submitted to storage under two conditions. The factors evaluated were temperature (25, 35 °C) and storage time (0, 30, 60, 90, 120, and 150 days), all samples were stored in low-density polypropylene packaging. All samples were evaluated for physicochemical properties, volatile profile, and bioactive compounds.

To choose the best processing condition for the guapeva jam, a 2^3 factorial design proposed by (Soares et al., 2021) was followed, in order to verify which would be the best formulation according to the sensory scores. Based on the results obtained previously, the parameters to be applied in this study were defined, guapeva pulp (40%), crystal sugar (60%), monohydrated citric acid (1%), and passion fruit albedo as a source of pectin (3%).

2.2 Physicochemical parameters

Physical composition of guapeva fruits

The fruits were submitted for physical analysis. The data relating to the length and diameter of the fruits were taken with the aid of a caliper, and the weights of the fruit, pulp, and endocarp were recorded after their separation with the assistance of balance analytics.

pH and titratable acidity

The determination of pH and titratable acidity for guapeva pulp and jams were carried out according to the Association of Official Analytical Chemists (2005).

Soluble solids and water activity

Soluble solids were carried out directly using an AKSO model RHBO-90 analog refractometer. The presence of syneresis in guapeva jam was determined by gravimetry, according to Khouryieh et al. (2005). The water activity (aw) was determined using the Aqualab CX-2 equipment in an environment with a controlled temperature of 25 ± 0.5 °C.

Color evaluation

The color analysis was performed at 25 °C using a digital colorimeter (Minolta CR4000, light source D65 in color space L *, a *, b *, chroma, and Hue in the CIELAB system). Calibration was performed with a white plate, following the manufacturer's instructions. The results were expressed in L * (luminosity), which ranges from 0 (black) to 100 (white); chroma (color saturation or intensity; where 0 represents the impure color and 60 the pure color); and hue angle (color angle; where 0º represents the red, 90º the yellow; 180º the green, 270º the blue, and 360º the black). All analyzes were performed in triplicate with three replications.

Sugar contents

For sample preparation, 1 g of pulp and jam were weighed and then diluted in 10 mL of ultrapure water (Milli-Q). Then, the samples were placed in an ultrasonic bath for 30 min at room temperature. After this process, the sample was centrifuged, and the supernatant was collected. Then, it was filtered through a PTFE (Polytetrafluoroethylene) hydrophilic filter with a pore size of 0.22 µm and kept under refrigeration until the moment of analysis (Warthesen & Kramer, 1979).

The determination and quantification of the carbohydrate profile were performed by high-performance liquid chromatography - HPLC, under the following analysis conditions: Agilent equipment, model 1260 infinity II, equipped with a refractive index-RID detector at 40 °C, using a column chromatographic Supelcogel C-610H (30 cm × 7.8 mm) – Sigma-Aldrich, pre-column Supelguard C610H (5 cm × 4.6 mm) – Sigma – Aldrich, the mobile phase using deionized water, with the flow (mobile phase flow rate) of 0.5 $mL.min^{-1}$, the total run time of 18 min and oven temperature of 40 °C. Glucose, fructose, sucrose, xylose, cellobiose, and arabinose were used as a standard to identify the compounds by comparing their retention time. The chromatographic method used was according to the methodology of the Supelcogel Column Application Manual - Sigma Aldrich.

Lipid profile of guapeva pulp

Pulp samples of guapeva were encapsulated with 24% maltodextrin due to high humidity and lyophilized (Liotop, model L101). The samples were subjected to extraction of crude oil, according to Santos et al. (2015), using hexane as the solvent in a soxhlet extractor. After oil extraction by soxhlet, the fatty acids were analyzed with the identification of methyl esters. For fatty acid identification and quantification analysis, 0.2 gram of the extracted oil was saponified with sodium hydroxide and methanol solution by reflux for 30 min (Hartman & Lago, 1973). Then, the oil was dissolved in a cryogenic tube with a capacity of 2 mL, 1 mg of the oil in 100 µL of a solution of sodium hydroxide (NaOH) 1 mol L^{-1} in ethanol/water (95:5). Then, the oil was homogenized and hydrolyzed in a microwave oven at 30%

power for 4 min. After cooling, 400 µL of 20% hydrochloric acid (HCl), 1 g of NaCl, and 600 µL of ethyl acetate were added and vortexed for 10 s; this mixture was allowed to stand for 5 min. Next, an aliquot of 300 µL of the organic layer was removed, placed in a microcentrifuge tube, and dried by evaporation, thus obtaining the free fatty acids. After the evaporation process, the free fatty acids were methylated with 100 µL of BF 3 (boron trifluoride-methanol solution) by heating for 10 min in a water bath at 60 °C, extracted in 500 µL of hexane, and fractionated by gas chromatography.

The profile of methylated fatty acids was analyzed in a Gas Chromatograph HP7820A (Agilent) 91 equipped with a flame ionization detector at the Chromatography Laboratory, Department of 92 Chemistry, Federal University of Minas Gerais. A Supelcowax-10 30 m × 0.2 mm × 0.2 µm column 93 (Supelco) was used. The column was held at 150 °C for 1 min and ramped to 260 °C at 10 °C/min; 94 injectors (1/20 split) at 250 °C and detector at 260 °C. Hydrogen was employed as carrier gas (6 95 mL/min) with an injection volume of 1 µL. FAME composition was compared with the standard 96 FAME C14-C22 (Supelco nº 18917).

Characterization of bioactive compounds

The methodology proposed by Oliveira et al. (2011) proposed to prepare the extracts, where 1 g of the sample was weighed and diluted in 10 mL of the solvent (70% Ethanol v/v). According to the adapted Folin-Ciocalteu method proposed by Waterhouse (2002), total phenolic content was determined. Results were expressed as mg of gallic acid equivalents (GAE).100 g^{-1}. The complete carotenoid range was determined according to the methodology described by Higby (1962). Results were expressed as mg of total carotenoids 100 g^{-1} of the sample. The vitamin C content of jam was determined by a colorimetric method with 2,4-dinitrophenylhydrazine (2,4-DNPH), according to Strohecker & Henning (1967). Results were expressed as mg of ascorbic acid. 100 g^{-1} of the sample.

The determination of the DPPH free radical scavenging capacity (2.2 diphenyl-1-picrylhidrazil) was estimated by the protocol developed by Brand-Williams et al. (1995) and adapted by Rufino et al. (2007). Briefly, a solution of DPPH (600 lM) was diluted with ethanol to obtain an absorbance of 0.7 ± 0.02 units at 517 nm. Then, the jams extracts (0.1 mL) were allowed to react with 3.9 mL of the DPPH solution for 30 min in the dark, and the decrease in absorbance (ABS) at 517 nm was monitored with a spectrophotometer. The results were expressed in EC50, which is the ability to reduce the initial DPPH concentration by 50% (g sample/g DPPH). Furthermore, antioxidant activity was determined in all jams in ethanol extract 80% by the iron reduction method (FRAP - Ferric Reducing Antioxidant Power), according to Benzie & Strain (1996) with modifications by Rufino et al. (2006). The readings (ferrous tripiridyl triazine complex) were measured on a spectrophotometer at 595 nm, and the results were expressed in µmol $FeSO_4$/g jam and pulp.

Texture profile of guapeva jam

The Texture Profile Analysis (TPA) was performed according to Friedman et al. (1963) using a Texturometer TA-XT2, with a 20 mm diameter flat-bottom stainless-steel cylindrical probe. Hardness, adhesiveness, cohesiveness, elasticity, gumminess, and chewability were assessed from force/time curves obtained using the Texture Expert Version 1.22 program for TPA.

Characterization of the volatile compounds of guapeva jam

The analysis was carried out under operating conditions: a fiber of polydimethylsiloxane/divinylbenzene (PDMS/DVB) 65 µm was used to partition volatile compounds present in the sample. Before use, the fibers were conditioned at a temperature of 300 °C for 1 h. Then, the samples (fiber plus jam sample) were stored for 30 min at 40 °C under the agitation of 250 rpm. Next, each fiber was exposed to a 1cm space in the head of the 10 mL glass bottle and exposed to 70 °C; after this, the syringe was automatically transported to the GC–MS injector, in which the volatile compounds were desorbed at 250 °C for 2 min in the splitless mode. A capillary column of silica (30 m × 0.25 mm and 0.25 µm thick) was used with a stationary phase of 5% diphenyl and 95% polydimethylsiloxane (DB5). The temperature of the injector was 270 °C, and the column was programmed to have an initial temperature of 60 °C, with 3 °C added every minute until the temperature reached 270 °C. The carrier gas (helium) flow rate was 1.8 $mL.min^{-1}$ in splitless mode, with an initial column pressure of 100 kPa. The mass spectrometer (MS) conditions were selective mass detector operating by electronic impact and impact energy of 70 eV; scanning speed 1000 $m/z.s^{-1}$; scan interval of 0.5 fragments. s^{-1} and detected fragments of 29Da and 600Da. The compounds were identified by comparing the mass spectra and retention indices using the Willey 8 libraries and specific literature (Adams, 2007), using an alkane pattern (C5-C20) as the base for calculation.

2.3 Statistical analysis

For the fruit characterization, the results were expressed as mean values ± standard deviation of the analyzes performed in triplicate and were treated using the Statistica 7.0 software. For the analysis of the jams, physicochemical and bioactive compounds were submitted to variance analysis ($p \leq 0.05$). In the case of statistical significance, the Tukey test was evaluated at the $p \leq 0.05$ confidence interval, with a 95% confidence level, using the SISVAR software. Regression analysis was used to explain the possible changes caused by the influence of time and temperature, and the model with the best fit was chosen through the coefficient of determination (r^2).

3 Results and discussion

3.1 Fruit characterization

Physical characteristics results show that the guapeva fruit tends to have a pulp yield of 24.4 ± 0.02, with an average weight of 77.85 g ± 0.35, spherical shape, and longitudinal and transversal diameters of 6.7cm ± 0.11 and 4.58cm ± 0.14, respectively. The pulp had a color angle of 80.05° ± 2.12, tending to yellow

color. The values found for luminosity (L*) and chromaticity (C*) were 30.79 ± 2.89 and 16.28 ± 1.82, respectively. Regarding acidity, the pulp showed 0.70 g.100 g^{-1} and pH 5.9 ± 0.05, which classified the flavor as moderately acid. In addition, the pulp showed a result of 24.53 °Brix for total soluble solids (Table 1). Fruits with high soluble solids contents are more appropriate for industrial processing and may have a higher yield, less processing time, and lower operating costs (Santos et al., 2018). The sugar profile of guapeva (Table 1) was formed mainly by sucrose (16.6 mg. mL^{-1}), followed by fructose (12.7 mg. mL^{-1}), and glucose content (4.7 mg. mL^{-1}) that confers sweetness to the fruit. The high sucrose content of this fruit can influence gel formation, making products such as sweets and jams more rigid (Menezes et al., 2009).

The vitamin C content obtained for guapeva (104 mg of ascorbic acid per 100 g of the sample - Table 1) was higher than the daily recommended value for an adult, which corresponds to 45 mg of vitamin C, based on Food and Agriculture Organization (2001). The consumption of foods rich in vitamin C can help develop and maintain the body, acting in collagen production, wound healing, iron absorption, and reducing susceptibility to infections (Cunha-Santos et al., 2019).

Table 1. Physicochemical composition, bioactive parameters, and carbohydrate profile of guapeva pulp.

Parameters	*Pouteria* cf. *gardneriana Radlk* pulp
pH	5.9 ± 0.05
Soluble solids (°Brix)	24 ± 0.03
Titratable acidity (g.100 g^{-1})	0.7 ± 0.02
*L**	30.79 ± 2.89
Chroma	16.28 ± 1.82
Hue	80.05 ± 2.12
Vitamin C (mg.100 g^{-1})	104 ± 0.49
Total carotenoids (mg.100 g^{-1})	2.5 ± 0.48
Total phenolics (**mg GAE.100 g^{-1}**)	514.6 ± 0.98
DPPH (**g of fruit. g^{-1} of DPPH**)	0.26 ± 0.06
FRAP (μmol of ferrous sulfate. g^{-1} of fruit)	136.9 ± 5.02
Carbohydrate profile	
Glucose (mg. mL^{-1})	4.7 ± 0.51
Sucrose (mg. mL^{-1})	16.6 ± 0.43
Fructose (mg. mL^{-1})	12.7 ± 0.11
FAME (%)	
Myristic acid (C14:0)	0.7
Palmitic acid (C16:0)	20.1
Palmitoleic acid (ω-7) (C16:1)	1.0
Stearic acid (C18:0)	9.3
Oleic acid (ω-9) (C18:1)	33.4
Linoleic acid (ω-6) (C18:2)	29.2
α- Linoleic acid (ALA, ω-3) (C18:3)	1.8
Arachidic acid (C20:0)	0.8
Others	3.8
Σ Saturated fatty acids	30.9
Σ Unsaturated fatty acids	65.4
Σ Monounsaturated fatty acids	34.4
Σ Polyunsaturated fatty acids	31.0
Ratio ω-6/ω-3	16.2

The carotenoid content found in guapeva was 2.50 mg. 100 g^{-1}. Therefore, carotenoids are natural oxidants and are one of the main constituents capable of significantly eliminating singlet oxygen. In addition, they protect lipid molecules, low-density lipoproteins, and DNA proteins against possible free radical attacks (Silva et al., 2014). In this sense when evaluating the methodological efficiency and amount of carotenoids found in the pulp of guapeva, we can infer satisfactory results for both parameters since guapeva has values close to pitanga (Lima et al., 2002) and cashew (Azeredo et al., 2006).

Regarding the capture of the DPPH radical expressed in EC50 by ethanol extraction, guapeva exhibited high antioxidant activity (0.26 g. g^{-1} DPPH - Table 1) because the lower the result obtained in EC50 is, the more significant is the efficiency of capture (Cottica et al., 2011). The total phenolic content for guapeva was 514.6 mg GAE.100 g^{-1} (Table 1), which classifies this fruit in the region of foods with high phenolic potential (Rufino et al., 2010). Besides this classification, guapeva for food processing can be justified by the presence of about 15 phenolic compounds in the guapeva pulp, including epicatechin and epigallocatechin, which were identified as principal components by Malta et al. (2013).

The fatty acid profile (Table 1) shows that the content of saturated fatty acids for the seed, peel, and pulp of guapeva was 36.1%, 45.6% 30.9%, respectively. The content of unsaturated fatty acids was around 62.9% for seed, 42.7% for peel, and 65.4% for pulp (Table 1). Considered the reference acid among vegetable oils (Monteiro-Silva, 2014), oleic acid (ω-9) was the acid with a more significant proportion for seed (46.1%) and guapeva pulp (33.4%) (Table 1). Therefore, the consumption of ω-9 is associated with reducing the adverse effects of diets composed of saturated fatty acids (Alves et al., 2014). Linoleic acid (ω-6) is an essential fatty acid because humans do not synthesize it, and this acid was found in the pulp (29.2%), peel (19.4%), and seed (15.4%) of guapeva (Table 1). Its α-linolenic isomer (ω-3) was also determined in the peel (3.4%), the pulp (1.8%,) and seed (0.9%) (Table 1).

3.2 Storage time and temperature effects on texture profile of the jam

Time and temperature significantly influenced the storage of guapeva jams ($p \leq 0.05$), showing an interaction between these two factors. It is possible to observe a gradual decrease in the soluble solids content caused by the action of the studied variables (Table 2). Even with the observed reduction, the soluble solids content is still following the recommended standard of the Codex Alimentarius (Food and Agriculture Organization, 2009), which says that this value should approach 60% in the case of typical fruit jams or preserves. Soluble solids are composed of water-soluble compounds, represented by substances such as sugars and organic acids (Santos et al., 2012). Thus, the decrease in the soluble solids in the present study may be associated with reducing the organic acid and the observed reduction of vitamin C content (Table 2).

Table 2. Physicochemical parameters, texture profile, colorimetric parameters, and sugar profile of guapeva jam during storage.

Parameters	Temperature (C°)	Time (Days)					
		0	30	60	90	120	150
pH	25 °C	7.15 ± 0.24^{Aa}	6.60 ± 0.01^{Ab}	6.53 ± 0.01^{Ac}	6.12 ± 0.01^{Ae}	6.46 ± 0.01^{Ad}	5.95 ± 0.02^{Af}
	35 °C	7.15 ± 0.24^{Aa}	6.60 ± 0.05^{Bc}	6.45 ± 0.01^{Bb}	5.83 ± 0.01^{Be}	6.11 ± 0.01^{Bd}	4.68 ± 0.02^{Bf}
Titratable acidity (g citric acid. g^{-1})	25 °C	2.46 ± 0.08^{Aa}	1.78 ± 0.01^{Bb}	1.18 ± 0.01^{Bf}	1.30 ± 0.07^{Be}	1.55 ± 0.07^{Bc}	1.46 ± 0.07^{Bd}
	35 °C	2.46 ± 0.08^{Ab}	1.78 ± 0.01^{Ad}	1.39 ± 0.01^{Af}	1.43 ± 0.07^{Ae}	2.03 ± 0.10^{Ac}	2.92 ± 0.10^{Aa}
Soluble solids (°Brix)	25 °C	67 ± 0.54^{Aa}	64 ± 0.51^{Ab}	58 ± 0.63^{Bd}	58 ± 0.53^{Bd}	58 ± 1.03^{Bd}	59 ± 0.63^{Bc}
	35 °C	67 ± 0.54^{Aa}	64 ± 0.83^{Bd}	62 ± 0.83^{Ae}	64 ± 0.41^{Ac}	66 ± 0.75^{Ab}	60 ± 0.51^{Ad}
Water activity (A_w)	25 °C	0.868 ± 0.10^{Ae}	0.931 ± 0.23^{Ba}	0.918 ± 0.61^{Ab}	0.890 ± 0.55^{Bd}	0.904 ± 0.12^{Ac}	0.832 ± 0.32^{Bf}
	35 °C	0.868 ± 0.12^{Ae}	0.933 ± 0.09^{Aa}	0.915 ± 0.28^{Bb}	0.893 ± 0.19^{Ad}	0.905 ± 0.11^{Ac}	0.845 ± 0.41^{Af}
Adhesiveness (N s)	25 °C	-260 ± 1.32^{Ae}	-371 ± 0.09^{Bf}	-197 ± 0.32^{Ac}	-202 ± 0.44^{Bd}	-23 ± 0.44^{Bb}	-08.50 ± 0.33^{Ba}
	35 °C	-260 ± 0.81^{Ae}	-320 ± 0.10^{Af}	-227 ± 0.81^{Bd}	-136 ± 0.51^{Ac}	-17 ± 0.12^{Ab}	-05.01 ± 0.16^{Aa}
Elasticity	25 °C	0.952 ± 0.05^{Ad}	0.918 ± 0.33^{Be}	0.957 ± 0.72^{Ac}	0.907 ± 0.11^{Bf}	0.969 ± 0.09^{Bb}	0.972 ± 0.33^{Ba}
	35 °C	0.952 ± 0.03^{Ac}	0.946 ± 0.81^{Ad}	0.90 ± 0.97^{Bf}	0.94 ± 0.09^{Ae}	0.975 ± 0.44^{Ab}	0.987 ± 0.19^{Aa}
Cohesiveness	25 °C	0.806 ± 1.01^{Ac}	0.706 ± 0.87^{Bf}	0.774 ± 0.54^{Bd}	0.718 ± 0.61^{Be}	0.895 ± 0.12^{Bb}	0.938 ± 0.11^{Ba}
	35 °C	0.806 ± 1.09^{Ad}	0.712 ± 0.66^{Af}	0.82 ± 0.37^{Ac}	0.743 ± 0.11^{Ae}	0.918 ± 0.09^{Ab}	0.956 ± 0.33^{Aa}
Gumminess (N)	25 °C	73.08 ± 0.53^{Ad}	87.30 ± 0.44^{Aa}	81.32 ± 0.39^{Ab}	78.11 ± 0.49^{Bc}	61.16 ± 0.41^{Ae}	50.29 ± 0.61^{Af}
	35 °C	73.08 ± 0.91^{Ac}	85.47 ± 0.91^{Bb}	67.64 ± 0.76^{Bd}	94.31 ± 0.71^{Aa}	53.63 ± 0.36^{Be}	48.42 ± 0.77^{Bf}
Chewiness (N)	25 °C	69.60 ± 0.23^{Ac}	80.01 ± 0.42^{Ba}	73.74 ± 0.82^{Bb}	69.42 ± 0.11^{Ad}	59.28 ± 0.62^{Ae}	48.89 ± 0.44^{Af}
	35 °C	69.60 ± 0.94^{Ac}	80.82 ± 0.41^{Ab}	88.61 ± 0.64^{Aa}	59.15 ± 0.09^{Bd}	50.23 ± 0.55^{Be}	47.77 ± 0.37^{Bf}
Hardness (N)	25 °C	90.62 ± 1.81^{Ad}	123.41 ± 0.33^{Aa}	113.3 ± 0.33^{Bb}	95.14 ± 0.01^{Ac}	68.37 ± 0.33^{Ae}	53.64 ± 0.37^{Af}
	35 °C	90.62 ± 1.62^{Ac}	119.97 ± 0.16^{Ba}	115.22 ± 0.48^{Ab}	80.09 ± 0.06^{Bd}	57.83 ± 0.74^{Be}	50.31 ± 0.91^{Bf}
*L**	25 °C	48.10 ± 0.55^{Aa}	25.13 ± 1.86^{Af}	39.20 ± 0.90^{Ab}	35.89 ± 1.04^{Ac}	31.58 ± 0.29^{Ad}	26.85 ± 0.20^{Ae}
	35 °C	48.10 ± 0.55^{Aa}	24.74 ± 0.28^{Bd}	37.28 ± 0.90^{Bb}	27.33 ± 0.54^{Bd}	30.84 ± 0.32^{Bc}	25.78 ± 4.29^{Bd}
Chroma	25 °C	19.05 ± 0.31^{Aa}	5.63 ± 0.15^{Bde}	11.46 ± 0.94^{Bb}	06.20 ± 0.49^{Bd}	7.26 ± 0.26^{Ac}	5.37 ± 0.06^{Be}
	35 °C	19.05 ± 0.31^{Aa}	6.33 ± 0.13^{Ad}	12.89 ± 0.74^{Ab}	11.83 ± 0.79^{Ac}	6.89 ± 0.45^{Bd}	7.01 ± 0.29^{Ad}
°*Hue*	25 °C	73.30 ± 0.60^{Aa}	64.48 ± 1.11^{Bbc}	59.10 ± 0.92^{Be}	61.56 ± 1.01^{Ad}	63.38 ± 0.32^{Ac}	65.90 ± 0.92^{Ab}
	35 °C	73.30 ± 0.60^{Aa}	67.87 ± 0.53^{Ab}	61.63 ± 0.77^{Ad}	61.98 ± 1.12^{Ad}	59.61 ± 0.48^{Be}	65.44 ± 1.08^{Ac}
Glucose (mg/mL)	25 °C	2.21 ± 0.12^{Ad}	2.46 ± 0.24^{Ab}	2.26 ± 0.11^{Ac}	1.97 ± 0.34^{Be}	1.96 ± 0.13^{Bf}	2.93 ± 0.33^{Ba}
	35 °C	2.21 ± 0.21^{Ad}	2.36 ± 0.31^{Aa}	2.28 ± 0.09^{Ac}	2.18 ± 0.31^{Ae}	2.03 ± 0.18^{Af}	2.30 ± 0.29^{Ab}
Fructose (mg/mL)	25 °C	6.35 ± 0.19^{Af}	7.43 ± 0.18^{Ad}	7.14 ± 0.07^{Be}	8.17 ± 0.29^{Ac}	9.07 ± 0.21^{Aa}	8.89 ± 0.44^{Ab}
	35 °C	6.35 ± 0.27^{Ae}	6.97 ± 0.21^{Bc}	8.99 ± 0.04^{Aa}	5.82 ± 0.18^{Bf}	6.70 ± 0.39^{Bd}	7.58 ± 0.32^{Bb}
Sucrose (mg/mL)	25 °C	35.25 ± 0.09^{Ac}	35.19 ± 0.37^{Ad}	36.50 ± 0.11^{Ba}	35.44 ± 0.02^{Ab}	27.72 ± 0.40^{Ae}	25.85 ± 0.21^{Bf}
	35 °C	35.25 ± 0.08^{Ac}	35.04 ± 0.31^{Ad}	38.04 ± 0.19^{Aa}	35.56 ± 0.09^{Ab}	27.98 ± 0.19^{Ae}	31.86 ± 0.07^{Af}
Total sugars (mg/mL)	25 °C	43.81 ± 0.17^{Ad}	45.08 ± 0.61^{Ac}	45.90 ± 0.09^{Ba}	45.58 ± 0.31^{Ab}	38.75 ± 0.17^{Ae}	36.67 ± 0.35^{Bf}
	35 °C	43.81 ± 0.14^{Ac}	44.37 ± 0.29^{Ab}	49.31 ± 0.11^{Aa}	43.56 ± 0.10^{Ad}	36.71 ± 0.32^{Af}	41.74 ± 0.20^{Ae}

Mean ± standard deviation (n = 3). a-f: in the lines, different letters indicate significant differences ($p < 0.05$); A-B: in the columns, different letters indicate significant differences ($p < 0.05$).

The water activity (aw) oscillated, tending to decrease with the end of storage from 0.868 to 0.832 and 0.845 for temperatures of 25° and 35 °C, respectively (Table 2). These results show that the time and temperature treatments significantly influenced variations in water activity in guapeva jams, demonstrating a correlation between the two variables ($p \leq 0.05$). This decrease is due to the hydrolysis of non-reducing to reducing sugars, which are more hygroscopic and depressants of water activity, in addition to being correlated with the reduction of moisture content according to the storage time (Riedel et al., 2015).

The texture of jams is directly related to gel formation, which depends on the concentration of acid, pectin, soluble solids, storage time, and temperature. A significant effect was observed at the level of 5% ($p \leq 0.05$) for the interaction between time and temperature in the parameters evaluated for the texture profile of guapeva jams (Table 2). In addition, the adhesiveness, elasticity, and cohesiveness of the jam produced in this work increased over the storage days (Table 2). However, an inverse behavior was observed when the jam's gumminess, chewiness, and hardness were analyzed. A decrease in parameters like hardness, chewiness, and gumminess during the storage period can reduce the soluble solids content observed for the guapeva jam (Table 2).

3.3 Storage time and temperature effects color and sugar contents

It was noticed that the colorimetric variables showed significant variations from the first month of storage (Table 2), especially in values of L* and Hue for the temperature of 35 °C. These variations indicate the tendency of specific color loss,

reducing the luminosity and changes from the initial yellow to reddish with the passing of the storage period. Such results suggest that no enzymatic oxidative processes occurred, resulting in the formation of compounds that confer a dark color, such as hydroxymethylfurfural and Maillard reaction (compound originated by the oxidation of vitamin C); or they may be related to the appearance of compounds resulting from the caramelization of sugar during storage; moreover, they can be associated with the formation of brown pigments by the Maillard reaction (Gabriel et al., 2015). Statistical analysis showed that the time-temperature interaction significantly affected the variables studied ($p < 0.05$).

Sugars determined for guapeva jam at time zero were glucose (2.21 mg. mL^{-1}), fructose (6.35 mg. mL^{-1}), and sucrose (35.25 mg. mL^{-1}). Over the storage period, an increase in glucose and fructose content was observed, with a consequent reduction in sucrose (Table 2). Statistical analysis ($p < 0.05$) revealed an interaction between time-temperature factors on the sugars individually. These results are possibly related to sucrose hydrolysis during storage, reducing sugars (glucose and fructose). This hydrolysis can be derived from interactions with reactions caused by organic acids since non-reducing sugars, such as sucrose, are hydrolyzed in acidic media (Oliveira et al., 2018), and it is essential to avoid crystallization. These results can also be justified due to the slight increase in the acidity of the samples from the ninetieth day of storage.

3.4 Storage time and temperature effects under the bioactive compounds

The interaction between time and temperature was not significant only for the total carotenoids of guapeva jam ($p < 0,05$), and time was the only variable that interfered in the results obtained for this constituent (Table 3). However, the time-temperature was statistically significant for the other bioactive compounds studied, such as total phenolics, vitamin C, DPPH, and FRAP (Table 3).

A decrease in the content of phenolic compounds during the storage for all temperatures studied was observed (Table 3). This degradation may be associated with the high instability of these compounds at temperatures between 20-40 °C since this temperature range can cause non-enzymatic reactions derived from the disruption of some cellular structures. Besides, the presence of oxygen caused using low-density polypropylene packaging may promote the more significant degradation of these compounds (Kamiloglu et al., 2015).

Reductions were observed in the carotenoid content during the 150 days of storage. It was also noted that the type of packaging used in the present study was not able to maintain the stability of this constituent ($p \leq 0.05$) (Table 3). Carotenoids were detected only until the fourth month of storage of the guapeva jams. As jam storage at temperatures below 30 °C and protection from light are essential factors for the stability of carotenoids (Brandão et al., 2018), the use of low-density polypropylene packaging and elevated temperatures may justify the behavior observed in this work for carotenoids in guapeva jams.

The vitamin C content was observed to decrease (Table 3), also being detectable only up to 120 days of storage, at the concentration of 11.85 mg ascorbic acid. 100 g^{-1} jam for 35 °C. According to Shinwari & Rao (2018), ascorbic acid is one of the thermolabile compounds most easily degraded by heat. Additional factors such as oxygen and light also interfere with its degradation. In this context, the high permeability of the packaging used in this work associated with high storage temperatures allows vitamin C degradation.

With the results obtained for DPPH radical reduction methodology, it can be seen that there was a significant difference ($p \leq 0.05$) in the antioxidant activity concerning the storage time and temperature, with a tendency to decrease. Considering that higher EC50 values correspond to less antioxidant activity, the results obtained show that guapeva jam has the potential to reduce the DPPH radical (Table 3). As observed for guapeva jam in this work, the reduction in antioxidant activity is related to the decrease in the values for ascorbic acid by heating and non-

Table 3. Bioactive parameters of guapeva jam during storage.

Parameters	Temperature (C°)	Time (Days)					
		0	30	60	90	120	150
Total phenolic content (mg GAE. g^{-1})	25 °C	133.75 ± 0.60^{Aa}	123.84 ± 1.26^{Ab}	106.33 ± 0.42^{Ad}	116.11 ± 0.32^{Ac}	107.36 ± 0.40^{Bd}	68.88 ± 0.35^{Be}
	35 °C	133.75 ± 0.60^{Aa}	115.66 ± 0.29^{Bb}	104.66 ± 0.58^{Bc}	92.29 ± 0.49^{Bd}	134.32 ± 0.84^{Aa}	114.83 ± 0.16^{Ab}
Total carotenoids (mg 100 g^{-1})	25 °C	1.21 ± 0.01^{Aa}	0.49 ± 0.01^{Ae}	0.85 ± 0.01^{Ab}	0.75 ± 0.02^{Ac}	0.52 ± 0.01^{Ad}	nd
	35 °C	1.21 ± 0.01^{Aa}	0.25 ± 0.02^{Be}	0.89 ± 0.02^{Ab}	0.74 ± 0.06^{Ac}	0.56 ± 0.01^{Ad}	nd
Vitamin C (mg of ascorbic acid.100 g^{-1})	25 °C	54.50 ± 0.11^{Aa}	45.03 ± 4.01^{Ab}	36.96 ± 0.74^{Ac}	26.83 ± 1.14^{Ad}	17.78 ± 0.80^{Ae}	nd
	35 °C	54.50 ± 0.11^{Aa}	40.73 ± 4.81^{Bb}	34.93 ± 0.04^{Bc}	26.62 ± 1.66^{Ad}	11.85 ± 0.11^{Be}	nd
DPPH (g jam/g DPPH)	25 °C	115.37 ± 1.40^{Ad}	84.62 ± 0.60^{Bf}	97.02 ± 0.32^{Be}	136.30 ± 0.94^{Bc}	161.26 ± 0.12^{Aa}	142.33 ± 0.52^{Bb}
	35 °C	115.37 ± 1.40^{Ac}	114.84 ± 0.23^{Ac}	100.95 ± 0.21^{Ad}	156.79 ± 0.46^{Ab}	156.75 ± 0.47^{Bb}	161.13 ± 0.12^{Aa}
FRAP (µM ferrous sulphate/g jam)	25 °C	46.94 ± 1.59^{Aa}	25.34 ± 0.19^{Bb}	15.35 ± 0.04^{Bc}	10.81 ± 0.02^{Bd}	9.59 ± 1.08^{Be}	7.48 ± 0.05^{Bf}
	35 °C	46.94 ± 1.59^{Aa}	37.23 ± 1.33^{Ab}	16.91 ± 0.26^{Ac}	14.20 ± 0.24^{Ad}	11.59 ± 0.21^{Ae}	8.96 ± 0.42^{Af}

Mean ± standard deviation (n = 3). Ascorbic Acid Equivalent (AAE) in milligrams/g sample. a-f: in the lines, different letters indicate significant differences ($p < 0.05$). A-B: in the columns, different letters indicate significant differences ($p < 0.05$). nd: non-detected.

enzymatic oxidation. Regarding the antioxidant activity by the FRAP method, the results consistently showed oscillations with a tendency to decrease. With the data in Table 3, it can be seen that the storage time and temperature significantly influenced the results obtained ($p \leq 0.05$) for the FRAP radicals.

It is worth mentioning that the presence of oxygen inside the packaging, which, together with other oxidizing agents present in foods, can accelerate the degradation of the bioactive compounds analyzed here (Jackman & Smith, 1996). In this way, an inert jam in the presence of oxygen would be the most suitable.

3.5 Storage time and temperature effects on volatile compounds

The analysis of the volatile profile of guapeva jams for the five storage times allowed the identification of 13 compounds experimentally (Table 4). The data for the volatile profile in Table 4 shows that initially, the jam presented fruity and herbal odors characteristic of hexanal and 2 – hexanal compounds from fresh fruit. In contrast, the jam had acid and acetic odors at the storage end (150 days). These results show that the aroma of guapeva jam is influenced by the storage time. In addition, some compounds are detected exclusively at each time, thus behaving like chemical markers of jam quality.

Results show that regardless of the storage time, the aroma of guapeva jam was composed predominantly of aldehydes. Among these volatile compounds, the main ones were hexanal and heptanal for aldehydes and acetic acid for acids (Table 4). Most of the compounds identified at time zero were aldehydes (61.5% of the total identified compounds), alcohols (26%), and furans (12.5%), maintaining this profile up to 30 days of storage (Table 4). Included in the compounds determined for time zero, those with the highest concentrations were hexanal (7.48%) and 2-heptanol (2.61%), and heptanol (2.72%). This behavior is possibly explained due to an increase in acidity and a reduction in pH, when a decrease in phenols was observed, plus a substantial increase in the relative concentration of C2 and C4s volatile acids and C5 aldehydes. Also, considering the results obtained for volatile compounds, it can be assumed that the increase in acidity in guapeva jams found in Table 2 relates to the formation of acetic and butyric acids.

For 60 days of storage, it was possible to observe an increase in the contents of aldehydes (35.5%) and acids (34%) and a decrease in the contents of alcohols (18%), furans (12%), and esters (7%) (Table 3). However, at 90 days, acids, aldehydes, furans, and alcohols represented 42, 40, 7.5, 7.0, and 3.5% of the total identified compounds. Many alcohols, aldehydes, and acids found in jams and fruits are derived from linoleic and linolenic acids (Ramadan et al., 2015). After the action of lipoxygenases, the hydroperoxyl-linoleic acid that results from

Table 4. Volatile compounds of guapeva jam at different storage times.

Compounds	R_t (min.)	Descriptor/ Odor	Temperature (°C)	Time (Days) (% area)					
				T0	T30	T60	T90	T120	T150
Ethanol	1.16	Alcoholic, sweet, and ethereal	25 °C	nd	nd	0.09	0.47	1.85	3.16
			35 °C	nd	nd	nd	0.133	1.62	2.27
2-Pentanol	3.09	Green	25 °C	2.61	2.33	1.51	0.31	0.09	nd
			35 °C	2.61	1.44	1.27	0.14	nd	nd
Acetic acid	1.49	Fruit and vinegar	25 °C	nd	nd	2.89	3.64	5.12	5.58
			35 °C	nd	nd	4.93	5.39	5.27	9.01
Pentanal	2.19	Cooked beef flavor	25 °C	nd	nd	nd	0.80	1.14	1.29
			35 °C	nd	nd	nd	1.13	1.47	1.61
Hexanal	3.67	Fruity	25 °C	7.48	5.46	2.29	1.73	0.93	nd
			35 °C	7.48	3.37	1.14	0.66	0.41	nd
2-Hexanal	5.81	Fruity	25 °C	2.59	2.47	1.19	1.12	1.51	0.92
			35 °C	2.59	3.09	2.33	1.84	1.66	1.37
Heptanal	6.09	Herbal and citric	25 °C	2.72	1.41	0.76	0.58	0.86	0.93
			35 °C	2.72	0.95	0.43	0.92	1.33	1.95
Ethyl hexanoate	6.26	Sweet, nut, and fruity	25 °C	nd	nd	0.03	nd	nd	0.31
			35 °C	nd	nd	0.24	0.13	0.28	0.27
Methyl hexanoate	6.80	Tropical fruity and pineapple	25 °C	nd	nd	nd	nd	0.34	0.27
			35 °C	nd	nd	nd	0.36	0.29	0.09
2-Pentylfuran	8.96	Sweet and green	25 °C	2.33	2.09	1.74	1.25	0.63	nd
			35 °C	2.33	1.66	0.94	0.37	nd	nd
2.6.10-Dodecatrien-1-ol	28.61	Floral	25 °C	1.62	1.27	0.19	0.09	nd	nd
			35 °C	1.62	0.91	0.12	nd	nd	nd
1.6.10-Dodecatrien-3-ol	28.70	Floral	25 °C	1.37	1.09	0.73	0.40	nd	nd
			35 °C	1.37	0.66	0.19	nd	nd	nd

T0, T30, T60, T90, T120, and T150: storage time 0, 30, 60, 90, 120, and 150; nd: non-detected a retention index.

this process undergoes hydroperoxide cleavage resulting in several products, including hexanal and nonanal, for example (Feussner & Wasternack, 2002). This fact corroborates with the present study since the presence of hexanal and n-hexanol were determined until 120 days (Table 4).

For 120 and 150 days of storage, it is possible to observe a decrease in aldehydes content and an increase in the contents of acids and alcohols (Table 4). Acids corresponded to approximately 42.0% of identified compounds, followed by 38.5% aldehydes, 13.5% alcohols, 3.5% esters, and 2.5% furans in 120 days. The decrease in the aldehyde content is evident in the time of 150 days, where the concentration of these components was 27%. Also, for the final storage time (150 days), the increase in the content of acids and alcohols made these components reach 51% and 17.5%, respectively. With these results, it is possible to assign acidity and alcoholic notes to the aroma of guapeva jam at the end of the storage period (150 days).

Despite the changes observed in the aroma of guapeva jam during storage, five components were present in all storage times, representing that the fundamental constitution of volatile compounds for this jam formed by 2-pentanol, hexanal, 2 - hexanal, heptanal, and 2-Pentylfuran.

4 Conclusion

Guapeva can be identified as a source of vitamin C, total phenolics, and high antioxidant activity, making it a raw material with high technological potential, suitable for the formulation of various products such as jams, juices, and nectar, etc. In addition, guapeva has expressive concentrations of ω9 fatty acids, followed by ω6 and ω3, indicating that these oils can offer health benefits during consumption. The guapeva jam was directly influenced by the interaction of time and storage temperature, being the time the variable that most interfered with the degradation and quality of the product, directly interfering with the bioactive compounds and the volatile profile. The temperature of 35 °C is less suitable for storage because of the potential to cause more significant degradation of the analyzed compounds. Initially, the guapeva jam presented fruity and herbal odors characteristic of hexanal and 2 - hexanal compounds from fresh fruit, and after 150 days, acid and acetic odors were predominant.

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