

Degradation kinetics of ascorbic acid from osmotically pre-treated passion fruit mesocarp and the study of its stability

Cinética de degradação do ácido ascórbico do mesocarpo do maracujá pré-tratado osmoticamente e o estudo da sua estabilidade

Cinética de degradación del ácido ascórbico del mesocarpio de maracuyá pretratado osmóticamente y estudio de su estabilidad

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Abstract

This research aims to evaluate the effect of temperature on the degradation of ascorbic acid from osmotically pre-treated passion fruit mesocarp and study its stability during storage. Passion fruit mesocarp slices were dehydrated osmoconvectively (60 °Brix/60 °C) and convectively dried at temperatures of 50, 60 and 70 °C until constant mass. During the drying process, degradation kinetics of the ascorbic acid were carried out and the experimental data was adjusted to match kinetic models of zero and first order. In addition, half-life, activation energy and the reaction coefficient (Q10) were calculated. Slices in natura were osmodehydrated for 90 days of storage and subjected to physical-chemical characterizations. Osmoconvective dehydration promoted a greater incorporation of total soluble solids (19 °Brix) and a lower percentage of water (58.51%) in the passion fruit slices. The drying process reduced the ascorbic acid content, but this compound was better preserved at a temperature of 60 °C. The first order kinetic model

proved to be more suitable to represent the degradation kinetics of ascorbic acid since it presented an activation energy of 83.33 kJ.mol⁻¹. During 90 days of storage, the samples achieved a reduction of 57.43% in ascorbic acid, remaining with a considerable content, 10.77% for the water content and pH below 4.5, making microbial growth impossible, consequently increasing the shelf life.

Keywords: *Passiflora edulis* Sims; Activation energy; Oxidation; Vitamin C.

Resumo

A presente pesquisa tem como objetivo avaliar o efeito da temperatura na degradação do ácido ascórbico do mesocarpo de maracujá pré-tratado osmoticamente e o estudo da sua estabilidade no decorrer do armazenamento. As fatias do mesocarpo do maracujá foram desidratadas osmoconvectivamente (60 °Brix/60 °C), em seguida submetidas a secagem convectiva nas temperaturas de 50, 60 e 70 °C até massa constante. No decorrer do processo de secagem foram realizadas uma cinética de degradação do ácido ascórbico e aos dados experimentais foram ajustados modelos cinéticos de zero e primeira ordem, além de se calcular o tempo de meia vida, a energia de ativação e o coeficiente de reação (Q10). As fatias in natura, osmodesidratadas e no decorrer de 90 dias de armazenamento, foram submetidas as caracterizações físico-químicas. A desidratação osmoconvectiva promoveu uma maior incorporação de sólidos solúveis totais (19 °Brix) e menor percentual de água (58,51%) as fatias do mesocarpo de maracujá. O processo de secagem promoveu redução no teor de ácido ascórbico, no entanto, este composto foi mais preservado na temperatura de 60 °C. O modelo cinético de primeira ordem, mostrou-se mais adequado para representar a cinética de degradação do ácido ascórbico, no qual apresentou uma energia de ativação de 83,33 kJ.mol⁻¹. Durante 90 dias de armazenamento, as amostras obtiveram uma redução de 57,43% de ácido ascórbico, ficando ainda com o teor considerável, o teor de água na ordem de 10,77% e o pH abaixo de 4,5 impossibilitando o crescimento microbiano, consequentemente aumentando a vida de prateleira.

Palavras-chave: *Passiflora edulis* Sims; Energia de ativação; Oxidação; Vitamina C.

Resumen

Esta investigación tiene como objetivo evaluar el efecto de la temperatura sobre la degradación del ácido ascórbico del mesocarpio de maracuyá pretratado osmóticamente y el estudio de su estabilidad durante el almacenamiento. Las rodajas del mesocarpio de maracuyá se deshidrataron osmoconvectivamente (60 °Brix/60 °C), luego se sometieron a secado por convección a temperaturas de 50, 60 y 70 °C hasta masa constante. Durante el proceso de secado, se realizó una cinética de degradación del ácido ascórbico y los datos experimentales se ajustaron con modelos cinéticos de cero y primer orden, además de calcular la vida media, la energía de activación y el coeficiente de reacción (Q10). Las lonchas in natura, osmodhidratadas y durante 90 días de almacenamiento, fueron sometidas a caracterizaciones físico-químicas. La deshidratación osmoconvectiva promovió una mayor incorporación de sólidos solubles totales (19 °Brix) y un menor porcentaje de agua (58,51%) en las rodajas de maracuyá. El proceso de secado redujo el contenido de ácido ascórbico, sin embargo, este compuesto se conservó mejor a una temperatura de 60 °C. El modelo cinético de primer orden resultó ser más adecuado para representar la cinética de degradación del ácido ascórbico, en el que presentó una energía de activación de 83,33 kJ.mol⁻¹. Durante 90 días de almacenamiento, las muestras obtuvieron una reducción del 57,43% en ácido ascórbico, quedando con un contenido considerable, el contenido en agua del orden del 10,77% y el pH por debajo de 4,5, imposibilitando el crecimiento microbiano, aumentando consecuentemente la vida útil.

Palabras clave: *Passiflora edulis* Sims; Energía de activación; Oxidación; Vitamina C.

1. Introduction

Brazil, is the world's largest producer of passion fruit, producing about 700 thousand tons of fruit annually, an average of 70% of this volume (peel and seeds) are considered to have no use and, therefore, are discarded by industries as they have no applications. The interest in its cultivation is due to the appreciation of its fruits, both for fresh consumption and in the form of industrialized products. Its fruit has a hard peel, yellow when ripe, and can also have a greenish-red or reddish color, the seeds are black and flat, surrounded by a gelatinous texture. It is popularly known for having relaxant properties and being a fruit rich in vitamins, mainly A and C. (Silva & Moura, 2010; Batista et al., 2017; Gonçalves et al., 2018; Bertani et al., 2019; Monteiro et al., 2019).

According to Monteiro et al. (2019), the passion fruit peel is rich in fibers such as pectin, which can be used for the manufacture of jams and sweets, and can also be used as flour, enriching products with dietary fibers, which helps preventing diseases. Soluble fibers, specifically, are being increasingly sought after for their reducing blood glucose activity, total cholesterol and triglyceride levels (Faleiro & Junqueira, 2016).

Technologies for the use of industrial waste allow to reduce the environmental impact, add value and enable the diversification of new food products with high nutritional value (Ronda et al., 2017; Thyberg and Tonjes, 2017; Matias et al., 2018). Osmotic dehydration as drying pre-processing has been widely used for reducing up to 50% of the water content contained in the food, giving the final product a better quality regarding appearance, flavor, aroma and texture (Goetten et al., 2016). According to Cardoso et al. (2018), the combination of drying techniques creates possibilities for new products with characteristics of the fresh product, conserving and improving the stability of the product during storage (Pessoa et al., 2011).

In this context, the present study aims to evaluate the effect of temperature on the ascorbic acid degradation from the osmotically pre-treated passion fruit mesocarp and to monitor its stability during storage.

2. Methodology

Passion fruit (*Passiflora edulis* Sims) were obtained in the city of Campina Grande, Paraíba considering their stage of maturation and visual quality. The whole fruits were subjected to an initial wash, sanitized with 50 ppm sodium hypochlorite for 5 minutes and rinsed to remove any remnant of the sanitizing solution. Using a stainless steel knife, the fruits were peeled, removing the epicarp, yellow film that covers the fruit and thus pulped, leaving only the mesocarp and the inner film which was removed by a stainless steel spoon. The mesocarp was then cut into slices. The samples were bleached (immersed in boiling water for 5 minutes) in order to inactivate the enzymes responsible for browning.

2.1 Osmotic dehydration and complementary drying

The passion fruit mesocarp slices were dehydrated osmoconvectively according to the best condition proposed by Silva Júnior et al. (2020) who, in their studies with osmotic dehydration of banana peels, optimized the conditions of 60 °Brix and 60 °C for 90 minutes. The slices were immersed in a dehydrating solution, in a plastic container having 60 °Brix of sucrose in distilled water, added with 30% passion fruit pulp, to incorporate the flavor of the samples. This container was taken to a greenhouse with air circulation, submitted to a temperature of 60 °C for 90 minutes.

The samples were placed in sieves to drain the dehydrating solution and placed on paper towels to absorb any remnant solution. The slices were placed on stainless steel screens forming a thin layer and taken to an oven with air circulation, at temperatures of 50, 60, and 70 °C until achieving constant mass.

2.2 Degradation kinetics of ascorbic acid

The degradation kinetics of ascorbic acid was performed in triplicate, weighing 1g of sample each time, in an initial interval of 5 and 5 minutes, and later, in 15, 60 and 80 minutes, for the three different drying temperatures (50, 60 and 70 °C) and their respective times. The determination of ascorbic acid was carried out according to the method of Tillmans (Brasil, 2008).

The reaction speed kinetic constants (k) of zero and first order were calculated according to Equations 1 and 2, respectively:

$$A = A_0 - k\theta \quad (1)$$

$$\ln \frac{A}{A_0} = -k\theta \quad (2)$$

Where: A is the concentration of the parameter evaluated after time " "; A₀ is the initial concentration of the evaluated parameter; k is the reaction speed constant; it's time.

To study the adjustments of the mathematical models to the experimental data, the computer program Statistica, version seven, was used. Non-linear regression analysis was applied by the Quasi-Newton method. The models were chosen considering the magnitude of the determination coefficient (R²).

Half-life ($\theta_{(1/2)}$) was calculated for the zero-order kinetic model (Equation 3) and for the first-order kinetic model (Equation 4). The k value used was from the kinetic model that best fit the experimental data on the degradation kinetics of ascorbic acid.

$$\theta_{(1/2)} = \frac{1}{k} \left(A_0 - \frac{A_0}{2} \right) \quad (3)$$

$$\theta_{(1/2)} = \frac{0.693}{k} \quad (4)$$

To verify the effect of temperature on the kinetic constant (k) on drying temperatures, the Arrhenius equation (Equation 5) was calculated according to Toralles et al. (2008). The k value used was that of the model that best fit the experimental data of the ascorbic acid degradation kinetics.

$$\ln k = \ln A - \frac{E_A}{RT} \quad (5)$$

Where: k is the reaction constant; A is the pre-exponential factor; E_A is the activation energy (minimum energy required for a reaction to start); R is the universal gas constant, 8.31 J mol⁻¹ K⁻¹; T is the absolute temperature (K).

The reaction coefficient (Q₁₀) which indicates the increase or decrease the reaction rate was determined by Equation 6.

$$Q_{10} = \frac{K_{(T+10)}}{K_T} \quad (6)$$

Where: K is the reaction constant; T is the absolute temperature (K).

2.3 Osmodhydrated mesocarp physico-chemical characterization and storage study

The slices of fresh yellow passion fruit mesocarp and osmodhydrated, in the best condition, 60 °Brix and 60 °C for 90 minutes, were studied in triplicate regarding the following physical-chemical parameters: pH, total titratable acidity, total soluble solids, content water and ash according to the methodology proposed by the Adolfo Lutz Institute (Brazil, 2008). The values of reducing, non-reducing and total sugars were determined according to the method of Lane and Eynon (1934). The ascorbic acid content was determined according to the Tillmans method (Brasil, 2008). Color parameters were determined using the Mini Scan Hunter Lab XE Plus spectrophotometer (Reston, VA, USA), in the Cielab color system which allowed obtaining the parameters: L* (luminosity); a* (transition from green (-a*) to red (+ a*)); and b* (transition from blue (-b*) to yellow (+ b*)).

The osmodhydrated samples submitted to complementary convective drying (60 °C) were stored in laminated packaging at room temperature. Each package contained 20g of mesocarp slices, undergoing physical-chemical analyzes (pH, total titratable acidity, total soluble solids, water content, ash, reducing, non-reducing and total sugars and ascorbic acid) at time zero and every 30 days, during 90 days of storage.

2.4 Statistical analysis

Statistical analyzes during storage were performed for experimental data in triplicate and the results went through the analysis of variance of single factor (ANOVA) of 5% of probability and the significant qualitative responses were submitted to the test of Tukey with the same level of 5% significance. For the development of statistical analyzes, Assistat software version 7.0 was used.

3. Results and Discussion

Table 1 shows the average results and standard deviations obtained in the physical-chemical characterization of the passion fruit mesocarp slices in natura and osmodeshydrated at 60 °Brix and 60 °C.

Table 1. Physico-chemical characterization of the passion fruit mesocarp in natura and osmodehydrated at 60 °Brix and 60 °C.

Parameters	<i>In natura</i>	Osmodehydrated
pH	6.01 ± 0.01	3.93 ± 0.006
Total titratable acidity (% citric acid)	0.063 ± 0.0015	0.128 ± 0.0008
Total soluble solids (°Brix)	2.8 ± 0.00	19.0 ± 0.00
Water content (% b.u)	93.83 ± 0.12	58.51 ± 1.21
Water activity (a _w)	0.993 ± 0.003	0.958 ± 0.001
Ashes (%)	0.33 ± 0.033	0.35 ± 0.034
Reducing sugars (% glucose)	0.25 ± 0.0025	2.00 ± 0.023
Non-reducing sugars (% sucrose)	0.09 ± 0.021	3.78 ± 0.21
Total sugars (% of glucose)	0.34 ± 0.02	5.78 ± 0.18
Ascorbic acid (mg/100g)	13.30 ± 0.52	9.21 ± 0.5
L*	60.35 ± 0.06	55.23 ± 0.023
a*	3.65 ± 0.04	0.59 ± 0.02
b*	20.53 ± 0.026	25.53 ± 0.12

Source: Authors.

The pH value of the slices in natura showed a non-acidic characteristic, with an average value of 6.01. The osmotic dehydration process promoted a decrease to 3.93, making them acidic. Carvalho et al. (2006) when performing osmotic dehydration of the yellow passion fruit mesocarp in sucrose solution obtained a lower value than the present study (3.87). The total titratable acidity showed a similar behavior with the pH, an increase in its average content from 0.063 to 0.128% of citric acid due to the acid characteristic of the osmodehydrated slices.

The osmodehydrated slices had a higher content of total soluble solids 19 °Brix compared to slices in natura. This behavior was expected due to the incorporation of solids throughout the dehydration process. Silva Júnior et al. (2020) obtained an increase from 5.73 to 16.05 °Brix when dehydrating banana peels at 60 °Brix and 60 °C.

The water content of the slices before the osmotic dehydration process was 93.83% and water activity 0.993, making the samples as highly perishable. Silva et al. (2019) in their studies with the passion fruit albedo, obtained water content of 93.55% and water activity of 0.995. Analyzing these same parameters at the end of the osmotic dehydration process, a reduction in water content to 58.51% and water activity to 0.958 was obtained. Products with high water activity are conducive to microbiological contamination, which justifies the need to apply additional drying.

The ash content for the fresh and osmodehydrated samples showed values greater than 0.30% and less than 0.40%. The highest value of 0.35% was obtained for osmodehydrated slices. Aragão et al. (2017) when applying the osmotic dehydration process in sword variety mango, they obtained the same values as the present study, being 0.38% for fresh mango and 0.48% for osmodehydrated mango. Paglarini et al. (2015), when evaluating the effect of osmotic dehydration conditions on the quality of araçá-pear raisins, obtained values between 0.24 and 0.48%.

The concentration of reducing sugars and total sugars resulted in values of 0.25% and 0.34%, respectively, which shows how low they are in the fresh passion fruit mesocarp. These values are considerable high for the product in natura due to the gain of solids in the osmotic process, the levels of reducing and totals sugar were 2.00% and 5.78%, respectively. Germer et al. (2011) when applying the osmotic dehydration process on peaches (55 °Brix/54.1 °C) obtained 2.003% for reducing and 9.47% for total sugars. Silva et al. (2017) in their studies with osmotic dehydration of pequi pulp flakes obtained total sugar

values of 2.77% for the fresh pulp and 4.31% when treated osmotically.

For vitamin C, the fresh and osmodehydrated sample presented the value of 13.30 mg/100g and 9.21 mg/100, respectively. Such considerable decrease is related to the heat sensitivity of the ascorbic acid. The values for luminosity (L^*), intensity of red (a^*) and yellow (b^*) were, respectively, 60.35, 3.65 and 20.53. They were quite substantial, depending on the physical characteristics of the sample, which has its color visually varying from white to yellow. The osmoconvective process reduced the luminosity (L^*) to 55.23, the red intensity (a^*) to 0.59 and increased the yellow intensity (b^*) to 25.53.

The osmodhydrated slices went through complementary convective drying at temperatures of 50, 60 and 70 ° C until achieving constant mass. The ascorbic acid degradation was evaluated over time. Table 2 shows the values obtained for the parameters of the zero order and first order kinetic models used to evaluate the degradation of ascorbic acid during the drying process.

Table 2. Kinetic parameters of ascorbic acid degradation at three different temperatures of osmoconvective yellow passion fruit mesocarp.

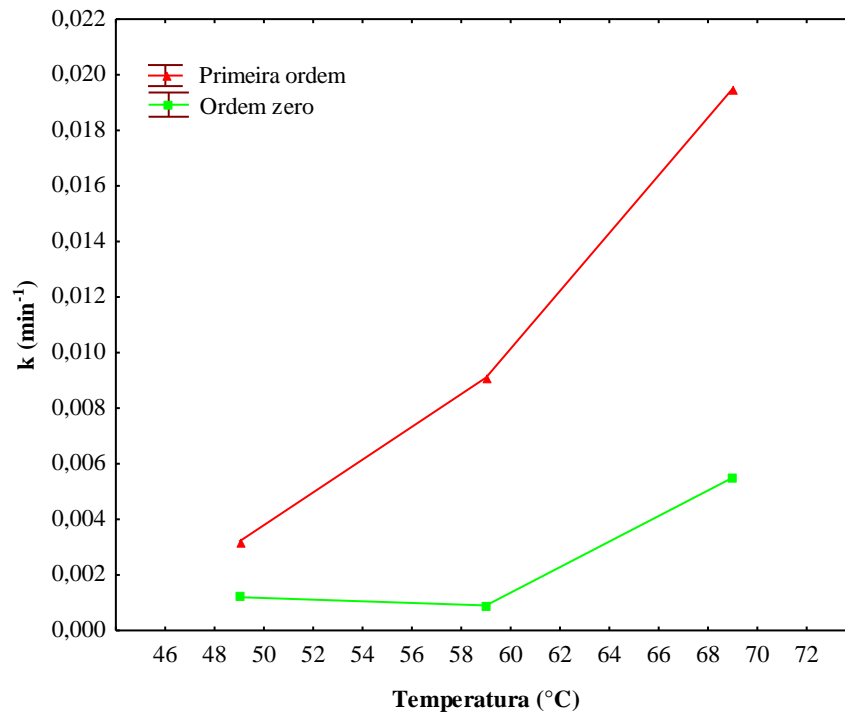
T (°C)	A ₀	Zero order		First order		$\theta_{(1/2)}$ (min)*
		k (min ⁻¹)	R ²	k (min ⁻¹)	R ²	
50	4.19	0.0012	0.9271	0.0032	0.9569	216.56
60	15.53	0.0009	0.8380	0.0091	0.9569	76.15
70	7.93	0.0055	0.7157	0.0195	0.8183	35.54

Note: * half-life calculated with the k value of the first order reaction. Source: Authors.

It can be seen from Table 2 that among the evaluated kinetic models the highest determination coefficients (R²) were obtained for the first order kinetic model. It shows how suitable a high R² is to represent the degradation of ascorbic acid (R²> 0.80). Fonseca et al. (2018) when evaluating the degradation kinetics of ascorbic acid in orange juice also concluded that the first-order model was best fitted the experimental data. Garbas et al (2003) studied the degradation of ascorbic acid in lyophilized plums. They also observed that the first order model was the one that best fitted the experimental data.

It is also observed, in Figure 1, the velocity of the constant k is temperature dependent. The ascorbic acid degradation rate varies with temperature. The higher the constant k value is, the greater the vitamin C degradation rate. Therefore, the temperature to which the product is exposed is considered an important factor for the degradation of ascorbic acid (Fonseca et al. 2018).

Figure 1. Speed constant k as a function of drying temperature for kinetic models of zero and first order.



Source: Authors.

Half-life was calculated from the velocity constant (k) of the first order reaction, since it had the best fit. Through the results it is observed that, with the increase in temperature, there was a reduction in the half-life time. So, the ascorbic acid present in the studied sample degrades more quickly at high temperatures. Table 3 shows the parameters of the Arrhenius equation and the reaction coefficient (Q10) for ascorbic acid degradation in the osmoconvective passion fruit mesocarp samples.

Table 3. Arrhenius equation parameters and reaction coefficient (Q10) for the ascorbic acid degradation in the mesocarp of yellow passion fruit.

T (°C)	Arrhenius Parameters			Q10	
	lnA	E _a (kJ mol ⁻¹)	R ²	(50-60 °C)	(60-70 °C)
50-70	25.32	83.33	0.994	2.84	2.14

Source: Authors.

The degradation kinetics showed an activation energy of 83.33 kJ.mol⁻¹. The Q10 value was 2.84 and 2.14, for a variation of 50-60 °C and 60-70 °C, respectively; Q10 value is higher when there is less loss of ascorbic acid. In this case, within the 50-60 °C range. Oliveira et al. (2013) obtained similar results studying the degradation kinetics and shelf life of whole mango juice, a Q10 value of 2.60.

The amount of ascorbic acid in the osmodehydrated passion fruit mesocarp slices before the drying process was 9.21 mg/100g. At the end of the drying process, when the samples reached hygroscopic balance, they showed the following concentrations of ascorbic acid: 2.53 mg/100g (50 °C), 11.12 mg/100g (60 °C) and 4, 26 mg/100g (70 °C). Through osmotic dehydration there was a higher solute (sucrose) incorporation into the slices, forming a protective film, which was able to decrease the ascorbic acid loss in samples set to a temperature of 60 °C. In addition, the water loss from the samples during

convective drying (60 °C) was found to have a water content of less than 12%, which favors ascorbic acid conservation in the dry product. Thus, taking into account that the ascorbic acid content is a fundamental parameter for the study of product stability, this compound was better preserved at a temperature of 60 °C, with 11.12 mg / 100g, expressing a loss of 16.39 % regarding the initial ascorbic acid content of the fresh product, which was 13.30 mg / 100g.

Table 4 shows the average values obtained for the physico-chemical characterizations of the mesocarp of passion fruit osmodehydrate (60 °Brix/60 °C) with complementary convective drying at 60 °C during 90 days of storage at 25 ± 2 °C.

Table 4. Physico-chemical characterizations of the osmodehydrated passion fruit mesocarp (60° Brix/60 °C) with complementary convective drying at 60 °C during 90 days of storage at a temperature of 25 ± 2 °C.

Parameters	Storage time (days)			
	0	30	60	90
pH	3.99 ^a	3.92 ^b	3.93 ^b	3.93 ^b
Total Titratable Acidity (% citric acid)	0.719 ^a	0.928 ^a	0.936 ^a	0.934 ^a
Total Soluble Solids (°Brix)	66.60 ^b	69.67 ^a	69.03 ^{ab}	67.13 ^{ab}
Water content (% b.u)	11.89 ^{bc}	15.23 ^a	13.03 ^b	10.77 ^c
Ashes (%)	0.79 ^b	0.74 ^b	0.81 ^b	1.05 ^a
Reducing sugars (% glucose)	10.27 ^b	9.00 ^d	9.74 ^c	10.95 ^a
Non-reducing sugars (% sucrose)	79.40 ^a	69.39 ^b	73.24 ^{ab}	73.30 ^{ab}
Total sugars (% of glucose)	89.67 ^{ab}	78.39 ^c	82.89 ^{bc}	86.25 ^{ab}
Ascorbic acid (mg/100g)	11.12 ^a	10.67 ^b	6.40 ^c	4.73 ^d

Note: Same lower-case superscript letters on the same row do not differ at the 5% probability level (p>0.05). Source: Authors.

During storage, pH value tended to be reduced while acid. However, from 30 to 90 days, the lower values were 4.0 with no statistical difference at the 5% probability level. The reverse behavior was observed with the values of total titratable acidity, 0.719 to 0.934% of citric acid, due to the reduction of the pH value during the 90 days of storage with no statistical difference.

For the soluble solids values, there is a variation between 66.60 to 69.67 °Brix, with no inclination for storage time increase. Between time 0 and 3, statistically, the samples showed significant differences. The water content during the storage time had an increase of 3.34% between the time zero and 30 days. Relevant reductions were observed for the time of 30 and 90 days, making it possible to verify that the product had a water content close to 12%, ensuring efficiency in preventing possible contaminating microorganisms.

At the end of storage, after 90 days, ashes were likely to have a 0.26% increase which can be verified by the incineration conditions, such as temperature variation and also depending on the composition of the food, which can be degraded or incorporated into the product over time. Statistically, through time zero and 60 days, samples had no significant difference (p > 0.05) and ash content was less than 1.0.

The levels of reducing, non-reducing and total sugars had no direct correlation with the storage time. The highest value of reducing sugars was 10.95% of glucose, observed at the time of 90 days with no significant differences (p > 0.05). For non-reducing sugars, there was a variation of 73.30 to 69.39% of sucrose over the storage period. However, only between zero and 30 days it showed statistical differences. The total sugars content did not differ when compared to the values obtained in time zero and 90 days, which were 89.67 and 86.25% glucose, respectively.

The results obtained make it possible to verify a considerable loss during storage varying by up to 6.39 mg/100g over

the 90 days. Statistically, the values calculated were significantly different for the 5% probability level ($p > 0.05$). The drying time is also an influential factor in the degradation of this parameter during the shelf life, even if it is the less detrimental factor. The loss was 57.46% in three months, considering the time zero and the final time of 90 days.

4. Conclusion

Osmoconvective dehydration promoted a greater incorporation of total soluble solids and a lower percentage of water in the slices of the passion fruit mesocarp. The drying process reduced the ascorbic acid content. However, this compound was better preserved at a temperature of 60 °C. The first-order kinetic model proved to be more suitable to represent the kinetics of ascorbic acid degradation, in which it presented an activation energy of 83.33 kJ.mol⁻¹. During 90 days of storage, the samples obtained a reduction of 57.43% in ascorbic acid, remaining with a considerable content. Water content in the order of 10.77% and the pH below 4.5, making microbial growth impossible, consequently increasing the shelf life.

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