Degradation kinetics of ascorbic acid in peach nectar during thermal processing

Cinética de degradación del ácido ascórbico en néctar de durazno durante el tratamiento térmico

Cinética da degradação do ácido ascórbico em néctar de pêssego durante tratamento térmico

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Abstract

Ascorbic acid is a beneficial component for health, but it is degraded during the thermal pasteurization of food products. The aim of this research was to determine the influence of temperature on the thermal degradation of ascorbic acid in peach nectar at 75, 85 and 95 °C, evaluating this effect at 0, 30, 60, 90 and 120 minutes. The degradation of ascorbic acid follows a first order reaction model with rate constants that vary between 5.5 to 10.9 x 10^{-3} min^{-1}. D-Values ranged from 211.28 to 418.73 min, while Z value was 69.4 °C. The values of the free energy of inactivation ranged between 112.63 and 117.17 $kJ.mol^{-1}$, while for the activation enthalpy the values varied between 25.37 and 25.54 $kJ.mol^{-1}$ and the range for the activation entropy was from -249.36 to -250.15 $J.mol^{-1}.K^{-1}$.It can be concluding that the reaction is endothermic and does not occur spontaneously. The knowledge of these values is important not only to explain the loss of ascorbic acid, but also to design and optimize thermal processes aimed at preserving the nutritional quality of peach nectar.

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Resumen

El ácido ascórbico es un componente beneficioso para la salud, pero es degradado durante la pasteurización térmica de los productos alimenticios. El objetivo de esta investigación fue determinar la influencia de la temperatura en la degradación térmica del ácido ascórbico en néctar de durazno a 75, 85 y 95 °C, evaluando el efecto a 0, 30, 60, 90 y 120 minutos. La degradación del ácido ascórbico se ajustó a un modelo de reacción de primer orden y las constantes de velocidad oscilaron entre 5,5 y 10,9 x 10^{-3}.min^{-1}. Los valores D oscilaron entre 211,28 y 418,73 min, mientras que el valor Z fue de 69,4 ºC. Los valores de la energía libre de inactivación estuvieron en el rango de 112,63 y 117,17 kJ.mol^{-1}. La entalpía de activación presentó valores entre 25,37 y 25,54 kJ.mol^{-1} y la entropía de activación osciló entre -249,36 y -250,15 J.mol^{-1}.K^{-1}. Se puede concluir que la reacción es endotérmica y no ocurre espontáneamente. El conocimiento de estos valores es importante no solo para explicar la perdida de ácido ascórbico, sino también para diseñar y optimizar procesos térmicos destinados a preservar la calidad nutricional del néctar de durazno.

Palabras clave: ácido ascórbico, cinética de degradación, parámetros cinéticos, néctar de durazno

Resumo

O ácido ascórbico é um componente de interesse por seus benefícios à saúde, porém é degradado durante a pasteurização térmica de produtos alimentícios. O objetivo desta pesquisa foi determinar a influência da temperatura na degradação térmica do ácido ascórbico em néctar de pêssego a 75, 85 e 95 °C, avaliando esse efeito em 0, 30, 60, 90 e 120 minutos. A degradação do ácido ascórbico foi ajustada a um modelo de reação de primeira ordem e as constante de velocidade variaram de 5,5 a 10,9 x 10^{-3}.min^{-1}. Os valores de D variaram de 211,28 a 418,73 min, enquanto o valor de Z foi de 69,4 ºC. Os valores da energia livre de inativação ficaram dentro da faixa de 112,63 e 117,17 kJ.mol^{-1}. A entalpia de ativação apresentou valores entre 25,37 e 25,54 kJ.mol^{-1} e a entropia de ativação variou entre -249,36 e -250,15 J.mol^{-1}.K^{-1}. Pode-se concluir que a reação é endotérmica e não ocorre espontaneamente. O conhecimento desses valores é importante não só para explicar a perda de ácido ascórbico, mas também para projetar e otimizar processos térmicos que visam preservar a qualidade nutricional do néctar de pêssego.

Palavras chaves: ácido ascórbico, cinética de degradação, parâmetros cinéticos, néctar de pêssego.

Introduction

Changes in eating habits are occurring at a faster rate, as consumers are more aware of the importance of nutrition in health and want to consume functional foods to improve their immune system and health in general (Plasek *et al.*, 2019).

Functional foods can be divided into three categories: conventional (such as citrus juices), modified (containing bioactive ingredients for enrichment or fortification) or food ingredients that are synthesized (such as inulin-type fructans, which provide prebiotic benefits) (Nwosu and Ubaoji, 2020).

Within this growing demand of functional foods, the global market for fruit and vegetable juices is projected to grow annually. Due to restrictions on consumption outside homes, many of these products are purchased for consumption at home, which benefits processing industries, which, faced with this growing demand, must avoid manufacturing practices that reduce the nutritional value of these beverages. In the case of citrus juices, this is largely related to their content of L-ascorbic acid (AA), which is a very reactive compound and therefore sensitive to physicochemical agents and environmental factors (Al Fata *et al.*, 2018).

However, during the production process, the juices usually receive thermal pasteurization to extend their shelf life, inactivating microorganisms and enzymes. This treatment is one of the causes of AA degradation, although other elements such as the pH of the matrix and photodegradation by UV radiation also contribute to it (Aguilar *et al.*, 2019; Cheng *et al.*, 2020). For this reason, AA is frequently added during industrial processing to prevent enzymatic browning and nutritionally enrich products, thus compensating for the aforementioned loss (Nowicka *et al.*, 2019).

Although some authors suggest that AA degradation during thermal preservation and storage of citrus juices may follow zero-order or first-order kinetics (NakilcioĞlu-Taş and Ötleş, 2020), the majority of published reports suggest that this process follows first-order kinetics (Peleg *et al.*, 2018). Kinetic degradation models constitute an effective method of predicting changes that may occur in physical and chemical parameters, which is why they are a valuable tool to control and improve processing and storage conditions (Zhang *et al.*, 2016).

During the processing of the product it is important to preserve, as much as possible, the sensory, nutritional and hygienic characteristics, but there is little information on the degradation of AA in peach nectar.

Although the kinetics of AA degradation in different fruit derivatives, especially citrus, has been studied, there is a lack of knowledge about the kinetic parameters of its thermal degradation in peach nectar. Knowing these values is of great importance from a practical point of view for the industries that produce this nectar, since it would allow a better control and optimization of the thermal processes, which in turn, would lead to a greater conservation of the nutritional quality of the product. Therefore, the objective of this research was to evaluate the influence of pasteurization temperature on AA degradation in peach nectar.

Materials and Methods

Determination of ascorbic acid content

The AA content was determined as reported by Nakilcioğlu-taş and Ötleş (2020) with modifications, through a colorimetric reaction. For this, an AA standard curve was used with solutions from 0.01 to 0.05 mg.mL^{-1} prepared from an AA stock solution (0.1 % w/v) containing pure AA and oxalic acid solution (0.4 % w/v). A mixture of 1 volume of distilled water and 9 volumes of 2,6-dichlorophenol indophenol solution was used as blank, while AA solution was used instead of distilled water for the standard curve. Absorbance was measured at 518 nm using a Shimadzu UV-Vis MINI-1240 spectrophotometer.

Collection and evaluation of samples

Peach nectar samples were taken for five days, at weekly intervals, at the entrance of the pasteurizer of an industrial production process, previously studied to verify that it was under statistical control.

Physicochemical analyzes were performed on the samples obtained to verify their similarity. The determinations made were:

Titratable acidity: expressed as citric acid, in percentage, evaluated by the volumetric titration method, using phenolphthalein as indicator (AOAC. 942-15, 1988).

pH: using a potentiometer with a glass electrode (Sartorius Mechatronics PP-5, Germany) (NTE INEN 389:1986).

Soluble solids: by means of a refractometer (PCE-018, Spain) with an interval of 0 to 18 °Brix (NTE INEN 380:1986).

Density: by pycnometry at 20 °C, expressed in $g.mL^{-1}$ (NTE INEN 391:1986).

Ascorbic acid degradation kinetics

The extracted samples were packed in amber glass bottles of 1 L capacity, which were placed in an ice water bath, for transport to the laboratory. For the study of the degradation kinetics, the samples were placed in closed test tubes, externally covered with aluminum foil to minimize the action of oxygen and light.

Were poured 10 mL of peach nectar into each tube and were accommodated in a thermostatic water bath (Memmert; Schutzasrt model; DIN 40050 – IP series, Germany). The investigated temperatures (representative of the pasteurization process) were 75, 85 and 95 °C. Once these temperatures were reached in the samples, the extractions were carried out every 30 minutes, for 2 hours. Five samples were available for each temperature-time condition investigated.

The degradation of AA at each temperature and at different treatment times can be defined with the following general differential equation for any reaction order:

$$(dC / dt) = \pm k\, C^n \quad (1)$$

For the zero order (n = 0) and the first order (n = 1), the following equations can be obtained, respectively:

$$C = C_0 \pm kt \quad (2)$$

$$\ln (C) = \ln (C_0) \pm kt \quad (3)$$

Where: C_0, is the initial concentration of AA ($mg.L^{-1}$); C is the AA concentration of compounds ($mg.L^{-1}$) after a certain process time; k is the reaction rate constant ($mg.L^{-1}.s^{-1}$ and s^{-1}, for n = 0 and n = 1, respectively); t is the process time (s) in seconds.

The kinetic constants obtained were fitted to the Arrhenius model in order to graphically determine the activation energy (Ea) of the process, by means of the slope of the representation of ln k as a function of 1/T, according to equation (4):

$$\ln (k) = \ln (A) - Ea/RT \quad (4)$$

Where: k, rate constant (whose u nits depend on the selected reaction order); A, pre-exponential factor; Ea, activation energy ($kJ.mol^{-1}$); R, universal gas constant ($J.mol^{-1}.K^{-1}$); T, absolute temperature (K).

For shelf life studies, the concept $t_{0.5}$ is usually used, which in this case represents the time it takes for the ascorbic acid concentration to reduce to half its initial value and can be calculated according to equation (5) for order zero:

$$t_{0.5} = C_0 / 2k \quad (5)$$

Or equation (6) for order one:

$$t_{0.5} = 0.693 / k \quad (6)$$

The D values (decimal reduction time at a certain temperature) and Z (temperature increase necessary to reduce the D value by a factor of 10) are terms usually used in the process of thermal inactivation of microorganisms to evaluate its effectiveness, but can also be used to determine the changes in concentration of some of the food components that occur during thermal processing. They can be calculated using the following expressions (Dhakal and Heldman, 2019):

$$D = 2.303 / k \quad (7)$$

$$\text{Log} (D_1 / D_2) = (T_1 - T_2) / Z \quad (8)$$

Another parameter of practical interest may be the temperature coefficient Q_{10}, which indicates how many times the rate of a reaction changes when the temperature changes by a value of 10 °C (Dhakal and Heldman, 2019). This value can be estimated using equation (9):

$$Q_{10} = (k_{T2} / k_{T1})^{10 / (T1 - T2)} \quad (9)$$

Thermodynamic analysis

The activation enthalpy ΔH^* ($J.mol^{-1}$), the free energy of inactivation ΔG^* ($kJ.mol^{-1}$) and the entropy of activation for vitamin C degradation ΔS^* ($J.mol^{-1}.K^{-1}$), at each temperature studied, were obtained using Eqs. (10), (11) and (12), respectively, as established by Martynenko and Chen (2016):

$$\Delta H^* = E_a - RT \quad (10)$$

$$\Delta G^* = - RT \ln (kh/k_B T) \quad (11)$$

$$\Delta S^* = (\Delta H^* - \Delta G^* / T) \quad (12)$$

where: Ea, is the Arrhenius activation energy ($kJ.mol^{-1}$); R, is the universal gas constant (8.314 $J.mol^{-1}.K$); T, is the absolute temperature (K); h is Plank's constant ($6.6262.\ 10^{-34}$ J.s); k_B is the Bolstzmann constant ($1.3806.10^{-23}\ J.K^{-1}$).

Statistical analysis

The results obtained are reported as the mean value and its standard deviation. The kinetic constants were calculated by linear regression and the adjustment of the predictive model through an analysis of variance, using Duncan's multiple range test ($p < 0.05$) to evaluate significant differences using the statistical program IBM SPSS (version 22), IBM, Armonk, New York, USA).

Results and discussion

The results of the characterization of the samples are presented in table 1.

As seen in table 1, there were no significant differences between the physical-chemical parameters of the samples analyzed for the kinetic study; therefore, it can be considered that the process operation was similar on the days the samples were obtained. The values obtained are similar to those found by Singh *et al.* (2016).

Ascorbic acid degradation kinetics

The AA degradation process depends on the combinations of time and temperature used during heat treatment or storage of the foods that contain it.

Table 2 shows the ascorbic acid content of the samples during their heat treatment .These values were adjusted by linear regression, using equations (2) and (3), to determine the most appropriate reaction order.

The results and kinetic data (for reaction order one) are shown in Table 3.

Table 1. Physical-chemical determinations of the peach nectar samples.

Parameter	Sample				
	1	2	3	4	5
Acidity (% citric acid)	0.31 ± 0.02 [a]	0.31 ± 0.02 [a]	0.31 ± 0.02 [a]	0.30 ± 0.02 [a]	0.30 ± 0.01 [a]
pH at 25ºC	3.61 ± 0.06 [a]	3.61 ± 0.05 [a]	3.66 ± 0.10 [a]	3.65 ± 0.10 [a]	3.64 ± 0.10 [a]
Soluble solids at 20 ºC (ºBx)	9.62 ± 0.10 [a]	9.66 ± 0.10 [a]	9.64 ± 0.20 [a]	9.62 ± 0.10 [a]	9.66 ± 0.20 [a]
Density at 25 ºC (g.mL^{-1})	1.030 ± 0.001[a]	1.031 ± 0.001[a]	1.030 ± 0.001[a]	1.030 ± 0.001[a]	1.031 ± 0.002[a]

Data are mean ± standard deviation of quintupled (n = 5). Means with the same lowercase superscripts within the same row are not significantly different ($p \leq 0.05$).

Table 2. Mean ascorbic acid (mg.100 g^{-1}) content at different times and temperatures

Temperature °C	Time (min)				
	0	30	60	90	120
75	62.9 ± 0.2[a]	54.4 ± 0.2[b]	44.1 ± 0.2[c]	37.7 ± 0.2[d]	33.1 ± 0.2[e]
85	58.5 ± 0.2[a]	40.7 ± 0.2[b]	35.3 ± 0.2[c]	27.4 ± 0.2[d]	23.7 ± 0.2[e]
95	53.7 ± 0.2[a]	30.4 ± 0.2[b]	24.6 ± 0.2[c]	17.5 ± 0.2[d]	13.8 ± 0.2[e]

Data are mean ± standard deviation of quintupled (n = 5). Means with the same lowercase superscripts within the same row are not significantly different ($p \leq 0.05$).

Table 3. Coefficients of determination (R^2) and kinetic data after heat treatments.

	R^2							
	Reaction Order		Kinetic data (for n=1)					
ºC	0	1	k (min^{-1})	$t_{0.5}$ (min)	D (min)	z (°C)	Ea (kJ.mol^{-1})	Q_{10}
75	0.9797	0.9940	0.0055	126.00	418.73			
85	0.9193	0.9737	0.0073	94.93	315.48	69.4	28.43	1.41
95	0.8689	0.9695	0.0109	63.58	211.28			

The percentage of AA loss was 47.4, 59.5 and 67.9 % for temperatures of 75, 85 and 95 °C, respectively, which is explained by its known thermosensitivity (Kadakal *et al.*, 2017). In an industrial pasteurization process, according to Petruzzi *et al.*(2017), treatment times range from a few seconds (High temperature-short time processes) to greater than or equal to 30 minutes (Mild temperature-long time processes), these losses will be highly variable. The influence of temperature on AA losses is more notable the longer the residence times are (Vieira *et al.*, 2015), as observed.

When the values of the ascorbic acid content were correlated with the treatment time, the highest values of the coefficient of determination (R^2) were found for the kinetics of order one. Based on the coefficients of determination obtained, the degradation of ascorbic acid in peach nectar follows first-order kinetics, and the values of the kinetic constant of the reaction rate increase with increasing temperature, which agrees with the results shown for citrus juices in the literature (Zhang, *et al.*, 2016; Dhakal and Heldman, 2019; Akyildiz *et al.*, 2021).

The values of k (table 3) found in this study are in the range of 0.0055 a 0.0109 min higher than the value 0.0025 min (Dhuique-Mayer *et al.*, 2007), but lower than the values between 0.004 y 0.015 min reported for rosehip nectar by other researchers (Kadakal *et al.*, 2017).

The importance of the food matrix in the degradation of ascorbic acid should be highlighted since the type of matrix will influence all the kinetic parameters. Which explains why, for example, the rate constant at 70 ºC can vary by a factor of approximately 25, between citrus juices and a fruit nectar from tropical forest trees or that during thermal processing of fruit juices the Q_{10} value ranges from 1.15 to 2 for ascorbic acid degradation (Dhakal and Heldman, 2019). Regarding the Z value expressed in ºC, very variable values have been reported, from 4.74 for a drink based on baobab fruit pulp (Abioye *et al.*, 2013), 48.99 in camu camu pulp (Calderón *et al.*, 2019), to 86.32 for orange juice (Akyildiz *et al.*, 2021).

The D value obtained decreases significantly with increasing pasteurization temperature, indicating that the ascorbic acid

degradation reaction is thermosensitive. In contrast, the Z value obtained (69.4 °C) is much higher than that reported for vegetative spores (5–12 °C) according to Peron *et al.* (2017), indicating that this reaction is less sensitive to temperature than the destruction of bacterial spores. Since the thermal processing of citrus derivatives and the degradation of ascorbic acid that occurs during the process are linked, knowing the value of D and Z of both processes would allow a better design of the pasteurization system.

In relation to the value of $t_{0.5}$, a great variability with temperature and food matrix has been reported. For example, in mango of Hilacha pulp it varies between 22.52 and 11.23 minutes for temperatures of 65 and 85 °C, respectively (Mendoza-Corvis, Hernández, and Ruiz, 2015), while it ranges between 87.72 and 96.3 days, depending of the method of pasteurization (Urquieta-Herrero *et al.*, 2021) in nance pulp. In orange juice (Akyildiz *et al.*, 2021) a decrease has been reported from 1136.3 to 666.5 s (58.65% of the initial value) for a temperature variation from 70 to 90 °C, slightly higher than the decrease percentage found in this work (50.46 %).

The Ea value is usually used to describe the energy required to reach the active state of vitamin degradation. In this research, the Ea value was calculated as the slope of the linear regression equation using equation (4). For the studied nectar, the Ea value was 28.43 $kJ.mol^{-1}$, a value similar to that reported by Akyildiz *et al.* (2021) and close to the lowest values reported in the literature. This indicates a very fast degradation since only a small activation energy barrier has to be overcome. However, great variability has been reported (Dhakal *et al*., 2018) in the results obtained by other researchers, with values between 21 and 128 kJ mol^{-1} for orange juice, which indicate the need to have values for specific products, considering factors such as the intrinsic characteristics of the product- variety and maturity, pH, concentration of solids and probably levels of dissolved oxygen- can alter the results of all the studied parameters.

Thermodynamic analysis

Table 4 shows the results of the thermodynamic variables for the loss of AA, calculated from equations 10, 11 and 12, at each of the temperatures studied.

Table 4. Enthalpy, free energy and entropy changes due to the thermal degradation process of ascorbic acid at different temperatures.

Temperature °C	ΔH^* ($kJ.mol^{-1}$)	ΔG^* ($kJ.mol^{-1}$)	ΔS^* ($J.mol^{-1}.K^{-1}$)
75	25.54	112.63	-250.15
85	25.45	115.10	-250.31
95	25.37	117.17	-249.36

The higher the value of the activation enthalpy, the higher the energy required for product formation. When this value is positive, it means that the reaction is endothermic and therefore requires a supply of energy for it to occur. The small decrease observed in ΔH* when increasing the temperature means that at higher temperatures, less energy is required to break the chemical bonds, therefore it is explained that the rate of AA degradation increases when the processing temperature increases.

The values found in the literature for ΔH*, differ widely, especially according to the characteristics of the product, and the temperature studied. For example, Remini *et al.* (2015), found a slight difference in the values of juice (fortified or not with different levels of ascorbic acid) of blood orange, in a relatively small range between 49–59 $kJ.mol^{-1}$. However, this value rose to 133 $kJ.mol^{-1}$ when the unfortified juice was subjected to deaeration.

On their research, Ordóñez-Santos and Martínez-Girón (2019) found for tree tomato juice (Ea = 41.27 $kJ.mol^{-1}$) ΔH* values of 38.72, 38.63 y 38.55 $kJ.mol^{-1}$ at temperatures of 70, 80 and 90 °C, respectively. While other researchers (Vieira *et al.*, 2015) have reported values of 18.32, 18.16 and 17.99 $kJ.mol^{-1}$ at temperatures of 50, 70 and 90 °C, respectively, with a value of Ea = 21.01 $kJ.mol^{-1}$. It is evident that the differences observed in the results of ΔH*, when working in the temperature range of industrial thermal pasteurization, is mainly attributable to the differences in the Ea values that the degradation reaction presents, typical of each product. In this study, the value of Ea (28.43 $kJ.mol^{-1}$) is intermediate between those of Ordóñez-Santos and Martínez-Girón (2019) and Vieira *et al.* (2015) and therefore the same occurs with the values of ΔH*.

The free energy change, ΔG*, indicates how spontaneous the process is through the difference between the activated and reactive states. For all the temperatures used in the thermal processing of peach nectar, positive values of ΔG* were observed, which shows that the degradation of ascorbic acid is not spontaneous. When these values are compared with those reported by other authors (Ordóñez-Santos and Martínez-Girón, 2019; Vieira *et al.*, 2015) it can be seen that the results obtained are intermediate between those of these researchers. Since the temperature range is similar, this can be mainly attributed to the differences in the values of k in equation 11.

When the entropy increases positively, this indicates that the system moves away from the state of thermodynamic equilibrium. In our study, the values found for ΔS* are negative, relatively little dependent on temperature in the thermal pasteurization range, which agrees with what is reported in the literature (Remini *et al.*, 2015; Ordóñez-Santos and Martínez-Girón, 2019; Cahyanti and Aminu, 2019) for the degradation of ascorbic acid, where greater importance of the characteristics of the product studied is indicated. The negative sign of ΔS* indicates that at the beginning of the degradation reaction, there is less molecular organization, thus confirming that it is not a spontaneous reaction.

Conclusions

This research was aimed at evaluating the changes in the AA concentration in peach nectar related to the pasteurization process of the product. The kinetic study showed that these changes follow first-order kinetics and that the increase in the rate constant is attributable to the increase in temperature. The results obtained represent a valuable tool to control and optimize the production conditions of peach nectar and increase its nutritional value.

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