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Effect of the Electric Field Frequency on Ascorbic Acid Degradation during Thermal Treatment by Ohmic Heating

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Abstract

In this work, the influence of the electric field frequency and solids content on the degradation kinetics of ascorbic acid during ohmic heating of acerola pulp and acerola serum was investigated. The degradation percentage of ascorbic acid in the pulp after 120 min of heating varied between 12 and 17%. For the serum, the degradation percentage was in the range of 13 and 18%. The results were fitted to the first-order model, and the kinetic rate constants ranged from 1.1 to $1.6 \times 10^{-3} \text{ min}^{-1}$ and from 1.1 to $1.5 \times 10^{-3} \text{ min}^{-1}$ for pulp and serum, respectively. D values ranged between 1480 and 2145 min for the pulp and between 1524 and 1951 min for the serum. A distinct behavior between the kinetic parameters of the pulp and serum in electric field frequencies ranging from 10 to 1000 Hz indicates that the presence of distinct amounts and types of solids might affect the rate of the electron transfer in electrochemical reactions. These variables may also affect the polarization process stimulated by the oscillating electric field. The non-achievement of the equilibrium of the polarization process may have an influence on oxidation reactions, affecting the predisposition to hydrogen donation from the ascorbic acid molecule.

Keywords

ohmic heating; ascorbic acid; electric field frequency; polarization; solids content

INTRODUCTION

Increasing consumer interest for nutritive fresh-like food products is driving the development of emerging processing technologies to ensure food safety and high nutritional and sensory properties. Ohmic heating (OH) is one alternative heat treatment where an

alternating current (AC) is passed through food materials, thereby leading to heat generation.^{1,2} This technology is particularly suitable for viscous products and liquid-particulate food mixtures because the heat is generated inside the food product.¹ It allows for processing at the rate of high-temperature, short-time (HTST) processes, but without the limitation of conventional HTST methods relative to heat transfer to particulates.²

For some years, it was presumed that the effects of the OH during food pasteurization and sterilization were solely thermal and that the applied electric field had no effect on biological cells and bioactive compounds.³ However, some studies have shown that non-thermal effects exist within the range of moderate electric field (MEF) strengths associated with OH.⁴⁻⁹ From this standpoint, the interaction of electric fields with food at the molecular, cellular, and tissue levels is important and needs to be evaluated. The effects of MEF treatments on bioactive components of foods have not been always considered, and the precise mechanism of MEF-induced changes is still not deeply understood.

In OH devices, the signal is an alternating bipolar current. When an electric field is applied to a sample, the molecules of the sample attempt to align themselves with the oscillating electric field in a process known as polarization. The electronic polarization is the displacement of electrons of atoms with respect to the nucleus, and the atomic polarization is the displacement of the atomic nuclei within the molecules.¹⁰ When a low-frequency alternating voltage is used, orientational movements of molecules that arise from the polarization process are slow and molecules have enough time to move and rotate in the field. However, when a high-frequency alternating voltage is applied, movements of molecules that arise from the polarization process become faster and may not be accomplished. The polarization depends upon the relaxation time of the molecular dipoles, which is, in turn, influenced by the molecular weight, mobility of the molecules, and temperature.¹¹

This work aimed to study the impact of MEF processing conditions during OH on the stability of bioactive compounds in food systems. Regardless of specific motivations or process objectives, it is of fundamental scientific interest to understand the kinetics of inactivation under various processing conditions, so that future processes may be designed with a full understanding of these effects. In a previous study, the effects of the electric field frequency on ascorbic acid degradation and color changes in acerola pulp were evaluated.¹² The objective of this study was to compare the degradation kinetics of the acerola pulp and the acerola serum to investigate the influence of the presence of solids when using different frequencies. Thus, our specific objectives were (1) to conduct experimental studies with acerola serum at different electric field frequencies (from 10 to 10⁵ Hz) and (2) compare these results to those previously reported by Mercali et al.¹² to obtain a better understanding of the influence of solids content on the precise mechanism of MEF-induced changes.

MATERIALS AND METHODS

Chemicals

Standard L-ascorbic acid with a purity of 99.7% and *meta*-phosphoric acid with a purity of 33.5–36.5% were obtained from Fisher Scientific Co. (Fair Lawn, NJ) and Sigma-Aldrich

(St. Louis, MO), respectively. Potassium phosphate monobasic was purchased from Sigma-Aldrich (St. Louis, MO). Deionized water was used throughout.

Acerola Pulp

Acerola pulp, supplied by EcoFruits Company (South Jordan, UT), was received frozen and was stored immediately at -18°C . The product had an initial vitamin C content of 1657 ± 45 mg/100 g.

Samples were thawed before each experiment by immersion in cold water. For acerola pulp, the experiments were conducted immediately after thawing. For the serum, thawed samples were then centrifuged (3000g at 5°C for 10 min) and the supernatant fraction was used immediately for conducting the experiments. The ratio between the serum and pulp was approximately 0.7 (w/w). The total solids contents of the pulp and serum were 12.8 and 9.9 g/100 g, respectively. The pulp was characterized as a high-acid product with a pH of 3.3 ± 0.3 . The initial temperature of the samples before starting the heating experiments was approximately $10\text{--}13^{\circ}\text{C}$.

OH Process

The OH apparatus used to conduct the experiments was comprised of a function generator (Tektronix, Inc., model AFG3252, Richardson, TX), a power amplifier (Industrial Test Equipment, model 500A, Port Washington, NY), an Agilent 34970A data acquisition unit and HP BenchLink Data Logger software (Agilent Technologies, Inc., Palo Alto, CA), a computer, and an ohmic cell. A more complete description of the OH setup can be found elsewhere.¹² The temperature was measured by two type-T thermocouples: one located close to the electrode and the other located away from the electrodes. The difference between the two measurements was lower than 2°C . The ohmic cell (9.9 cm of height) consisted of a 500 mL glass vessel with a water jacket. It had a lid to prevent evaporation from the product. The electrodes were made of titanium and curved to conform to reactor dimensions. The maximum interelectrode gap was 7.5 cm, and the minimum gap was 5.7 cm. The electrode height was 5.0 cm. The product was stirred using a magnet inside the cell and a magnetic stirrer plate (Fisher Scientific, Model Isotemp, Fair Lawn, NJ). The velocity of the agitation is an important factor to be considered because it has influence on the amount of oxygen incorporated in the product. In this manner, the velocity of the agitation of both products was matched to eliminate this variable during the experiments (500 rpm for the pulp and 350 rpm for the serum). Five different electric field frequencies (FQs) were studied: 10 , 10^2 , 10^3 , 10^4 , and 10^5 Hz.

The kinetic experiments were conducted at 85°C for up to 2 h. A critical consideration in such experiments (to ensure that an effect may be attributed to an electric field effect) is the need to maintain the same temperature history for both ohmic and conventional treatments. OH has its unique electrical heating mechanisms and temperature histories that are fundamentally different from the ones of the conventional process. To be able to match the time–temperature histories between electric field treatments and thermal controls, OH experiments were conducted using a controlled temperature cooling water in the jacket (in actual practice, it used 30°C and 75°C water in the jacket simultaneously). In contrast,

because conventional heat treatment lacked an internal energy source, the jacket was maintained at the test temperature (85 °C). This procedure allowed for the evaluation of the non-thermal effects of electricity because the temperature histories for conventional and ohmic treatments were the same. Sample plots of the thermal histories for OH and conventional heating (Figure 1) for acerola serum show that we were able to closely match temperature histories. Figure 1 represents the temperature measured close to the electrode. Time–temperature histories reported for acerola pulp showed a similar plot behavior.¹²

A model was developed to estimate the average electric field strength by solving Laplace's equation within the vessel domain. Figure 2 shows average electric field distribution within an ohmic cell when 30 V is applied. The electric field within most of the cell is reasonably uniform, except for the electrode edges. The model did not account for the presence of the magnet and agitation. The agitation itself may not make a lot of difference unless the shape of the liquid surface is greatly changed by the agitation. The magnet will alter the local field, but the average field will likely be quite unaffected. In this manner, the assumed field strength was considered realistic.

The time zero was set when the sample reached the desired holding temperature. Samples were withdrawn at various heating times (0, 20, 40, 60, 80, 100, and 120 min). Determination of ascorbic acid levels was carried out immediately after the heating experiments.

Conventional Heating Process

The conventional heating process was conducted in the ohmic cell without an electric field. The samples were heated by passing hot water through the jacket using a thermostatic water bath (Brookfield Engineering Laboratories, model EX-100, Middleboro, MA). The kinetic experiments were conducted at the same temperature (85 °C), and samples were collected at the same heating times ranging from 0 to 120 min.

Determination of the Ascorbic Acid Content

The ascorbic acid content was quantified using high-performance liquid chromatography (HPLC, Hewlett-Packard, HP Agilent 1100 system, Norwalk, CT), reverse C₁₈ Luna (5 µm, 250 × 4.6 mm, Phenomenex, Torrance, CA), and a diode-array detector (254 nm). The detailed methodology can be found elsewhere.¹²

Extraction of ascorbic acid was carried out adding *meta*-phosphoric acid solution to the samples. The mixture was homogenized for 30 s and centrifuged (1500g for 5 min). The supernatants were filtered through a 0.45 µm polytetrafluoroethylene (PTFE) membrane, diluted, and injected into the HPLC system. Samples were protected from light and kept on ice during all procedures.

Determination of the Kinetic Parameters

Traditionally, degradation of vitamin C in foods during thermal processing and storage has been described in terms of first-order kinetics and the Arrhenius equation.^{13–16} Because the data seemed to follow a simple linear decrease, the zero-order kinetics was also considered.

Then, a comparison between both models was carried out, and the model that better fit the experimental values was chosen to describe changes in ascorbic acid concentrations in acerola pulp during heating. The zero-and first-order models are given by

$$C=C_0-kt \quad (1)$$

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

where t is the time (min), k is the rate constant (min^{-1}), C_0 and C are the ascorbic acid contents at time zero and time t , respectively.

The decimal reduction time (D value), the time needed for a 10-fold reduction of the initial concentration at a given temperature, is related to k values according to eq 2.

$$D=\frac{\ln(10)}{k} \quad (3)$$

Statistical Analyses

Two independent experiments (replicates) were conducted for each frequency studied during OH and also for conventional heating. The results were fitted to the zero- and first-order models by linear and nonlinear estimation, respectively. The rate constants (k values) of all experiments were compared using the Tukey test (5% of the confidence level). Statistical analyses were performed using Statistica 7.0 (StatSoft, Tulsa, OK) and Microsoft Excel 2011 (MapInfo, Troy, NY).

The goodness of fit of the proposed models was evaluated by means of the χ^2 parameter, the determination coefficient (R^2), and the standard error of means (SEM). The average errors between the experimental values and the values predicted by the models (E) were also estimated.

RESULTS AND DISCUSSION

Degradation of ascorbic acid over time for acerola serum during conventional heating and OH (using different frequencies) is shown in Figure 3. Error bars have not been added because it would be impossible to distinguish between the different treatments. Standard deviations were lower than 3% for all experimental points. Figure 4 shows the results reported for acerola pulp.¹² The degradation percentage of ascorbic acid in acerola pulp after 120 min of heating varied between 12 and 17%. For the serum, the degradation percentage was in the range of 13 and 18%. These values were lower than expected on the basis of kinetic data reported in the literature.^{16–20} It is likely that the oxygen dissolved in the medium is the limiting reactant because the vitamin C content in acerola is very high, approximately 40 times higher than that of orange juice (1657 mg/100 g compared to 40 mg/100 g). Therefore, there is not enough oxygen in the medium to react with this extremely high vitamin C content. Another possibility is the degradation of some other antioxidants

(anthocyanins) that are competing for the available oxygen. According to de Rosso and Mercadante,²¹ the high ascorbic acid content is the main cause of the low stability of anthocyanin extracts from acerola. A recently published study performed in our laboratory evaluated the degradation kinetics of monomeric anthocyanins in acerola pulp during thermal treatment by OH and conventional heating.²² The anthocyanin degradation percentages after 90 min of OH at 75 and 90 °C were approximately 46 and 84%, respectively. This may be an indication that anthocyanin is degrading faster than ascorbic acid in acerola pulp. Another point that needs to be mentioned is that acerola has a very acidic pH (approximately 3.3), and at low pH, ascorbic acid is more stable.²³ When analyzing ascorbic acid degradation, it is important to consider that its stability is influenced by the intrinsic properties of the product and the process characteristics.^{13,15,18,20,24–27} Vitamin C degradation can occur through aerobic and/or anaerobic pathways, depending upon a number of factors, such as pH, acidity, metal ions, light, humidity, water activity, temperature, presence of amino acids, carbohydrates, lipids, and enzymes, among others.²³

From Figures 3 and 4, it can be observed that pulp and serum showed a distinct behavior when analyzing the degradation curves in the same range of frequencies. For a better comparison between treatments, the results of the ascorbic acid concentration against time were fitted to the zero-and first-order equations. The quality of the adjustment was evaluated through the statistical parameters presented in Tables 1 and 2. Both first- and zero-order equations proved to be suitable for modeling acid ascorbic degradation because the determination coefficients were above 0.98 for all treatments. When compared to the zero-order model, the first-order model yielded a better fit of the experimental data, with a smaller deviation between the values estimated by the model and experimental data and lower SEM and χ^2 values. The accuracy of the first-order model is confirmed by the small deviation between the values estimated by the model and the experimental data. Thus, the first-order model was chosen for predicting the ascorbic acid retention of acerola pulp and acerola serum during OH and conventional heating.

Table 3 presents the kinetic parameters of the first-order model obtained for each frequency evaluated during OH and also for the conventional process for both products. According to Mercali et al.,¹² the pulp showed kinetic rate constants ranging from 1.1 to $1.6 \times 10^{-3} \text{ min}^{-1}$ and D values ranging between 1480 and 2145 min. For the serum, the rate constants were between 1.1 and $1.5 \times 10^{-3} \text{ min}^{-1}$ and D values ranged from 1524 to 1951 min. Some points observed when comparing Figures 3 and 4 are confirmed by the statistical analysis. For acerola pulp, there was no difference ($p > 0.05$) between ascorbic acid degradation rates for experiments carried out with electric field frequencies ranging from 100 to 10^5 Hz. Also, the rate constants of these experiments did not differ from that found for the conventional heating treatment. The rate constant of the OH process at 10 Hz was higher than the rate constants of all other treatments. However, for the serum, a different behavior was observed. The rate constant of the OH process at 10 Hz was statistically similar to the rate constants of the treatments at 100 and 1000 Hz. These three conditions showed rate constants statistically different from the experiments conducted at 10^4 and 10^5 Hz. Also, the conventional process showed similar rate constants to the ones obtained for treatments using 10^4 and 10^5 Hz.

Higher degradation rates at lower frequencies may be explained by the occurrence of electrochemical reactions (electrolysis of water and electrode corrosion). Electrolysis of water releases hydrogen and oxygen in the medium, which may cause additional oxidation of ascorbic acid.²⁸ Electrode corrosion releases metal ions that also catalyze the oxidation process.²⁸ Corrosion of electrodes and apparent (partial) electrolysis of the heating medium with most types of electrodes at low-frequency ACs were noticed in experiments carried out by Samaranayake and Sastry.²⁹ These authors verified that apparent electrolysis was seen at all pH values, even though titanium electrodes showed a relatively high corrosion resistance.

These electrochemical reactions occur within a narrow area between electrodes and the product, which acts as a capacitor. When an AC voltage starts flowing through the system, charges start to build up in this area close to the surface of the electrode. If a low-frequency alternating voltage is applied, there is enough time to reach the Faradaic threshold potential and the layer becomes saturated with charges. After the saturation point, any current that flows through it results in electrochemical reactions.³⁰ On the other hand, when a high-frequency alternating voltage is used, the potential raise and reverse before it reaches the Faradaic threshold potential point as a consequence the electrochemical reactions do not take place.

The distinct behavior shown between acerola pulp and acerola serum at frequencies ranging from 10 to 1000 Hz indicates that the presence of different amounts of solids may have an influence on this process. Moreover, the serum and pulp have a distinct composition of solids: in the serum, most of the solids are soluble solids, such as sugar, vitamins, and acids, while in the pulp, there are also insoluble solids, such as fibers, starch, pectin, and long-chain carbohydrates. The amount of solids and even the predominant type of solid (insoluble or soluble solids) may have affected the drag for ionic mobility and, consequently, the rate of electron transfer. The particle type, size, concentration, shape, and orientation in the electric field may affect the mass transport processes by diffusion and/or convection that occur in the narrow layer between the electrodes and the product, which, in turns, influences the rate of the electron transfer reactions.

In high frequencies (10^4 and 10^5 Hz), ascorbic acid degradation was similar to the degradation found in the conventional heating. This behavior was observed for both acerola pulp and serum. This means that the electrochemical reactions were probably minimized and the main mechanism of degradation was the oxidation. Some authors recommend the use of high-frequency ACs to effectively suppress electrochemical phenomena at electrode–solution interfaces.^{2,28,31}

Another point that needs to be considered is the effect of the polarization stimulated by the oscillating electric field. The polarization process is critically dependent upon the relaxation time of the molecular dipoles. Relaxation time refers to the time taken by the dipoles to revert to random orientation when the field is removed and is influenced by their molecular weight, mobility, and temperature.^{11,32} Because liquids and solids have different relaxation times, the polarization of the whole sample depends upon the relative contribution of the phases present in the sample. The acerola pulp had higher solids content and more insoluble solids (fibers, starch, pectin, and long-chain carbohydrates) than the serum, which makes

them have different dielectric properties, and, consequently, different behaviors when subjected to electric fields.

The ascorbic acid is a small polar water-soluble molecule.³³ Even though its molecular weight is higher than water, some considerations can be performed comparing its behavior to the water molecule. When an electric field is applied at low frequencies to a polar liquid (such as water), the time interval taken for the field to reverse its polarity would presumably be longer than the relaxation times of the molecules. This means that there is enough time available for the molecules to respond and reorient in accordance to the direction of field changes. As the field frequency increases, the time interval between reverse movements in field polarity gradually becomes of a similar order to the relaxation times of the molecules. At this point, they still respond to the changing fields but with increasing lag time and energy absorption. At very high frequencies, the dipole motion can no longer keep up with the changing field. These molecules will cease to respond and remain in their random, steady-state orientations.^{11,32}

However, in many materials, such as in acerola pulp, the water molecules are often chemically bound to other molecules in the material. The ascorbic acid is also chemically bound to water and other substances because of the presence of hydroxyl groups in its structure. According to Ryyänänen,³⁴ hydrogen bonds restrict the free movement of the molecules, and therefore, bound molecules possess longer relaxation times. In the same way, the extensive intermolecular bonding interactions prevalent in solid materials restrict dipolar orientations.¹¹ In this manner, particle type, size, concentration, and polarity may affect bonding interactions and, consequently, influence the achievement of the equilibrium of the polarization process. These phenomena may have influenced the oxidation reactions during heating of acerola pulp and serum, affecting the predisposition to hydrogen donation from the ascorbic acid molecule.

This study highlights the importance of evaluating dielectric properties of food products to determine their behavior when subjected to an electric field. The increase in the use of electrical heating methods in recent years requires the knowledge of electrical properties and their effects on food processing. Further work is necessary to better understand the effects of the electric field frequency during OH.

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Figure 1. Time-temperature histories (the first 25 min of heating) of the ohmic and conventional treatments for acerola serum, showing closely matched thermal histories.

Figure 2. Average electric field distribution within the ohmic cell (cross-section) when 30 V is applied.

Figure 3. Degradation of ascorbic acid in acerola serum during OH and conventional heating processes. The standard deviations were lower than 3% for all experimental points.

Figure 4. Degradation of ascorbic acid in acerola pulp during OH and conventional heating processes. The standard deviations were lower than 3% for all experimental points. Adapted with permission from ref 12 (Copyright 2013 Elsevier, License number 3305350538453).

Statistical Parameters Evaluating the Zero- and First-Order Model Performance To Describe Ascorbic Acid Degradation during OH and Conventional Heating of Acerola Pulp

Table 1

process	FQ (Hz)	zero-order model				first-order model			
		R ²	E (%)	χ ²	SEM	R ²	E (%)	χ ²	SEM
OH ^a	10	0.99	7.34	6.8 × 10 ³	1.3 × 10 ⁴	1.00	0.34	2.1 × 10 ⁻⁵	3.9 × 10 ⁻⁵
OH	10 ²	0.99	5.66	2.7 × 10 ³	5.0 × 10 ³	0.99	0.40	1.9 × 10 ⁻⁵	3.5 × 10 ⁻⁵
OH	10 ³	0.98	10.63	8.9 × 10 ³	1.7 × 10 ⁴	0.98	0.48	3.9 × 10 ⁻⁵	7.3 × 10 ⁻⁵
OH	10 ⁴	0.99	7.85	4.8 × 10 ³	9.1 × 10 ³	0.99	0.55	3.4 × 10 ⁻⁵	6.5 × 10 ⁻⁵
OH	10 ⁵	1.00	2.32	6.0 × 10 ²	1.1 × 10 ³	1.00	0.22	7.9 × 10 ⁻⁶	1.5 × 10 ⁻⁵
CH ^b	0	0.99	5.12	3.6 × 10 ³	6.8 × 10 ³	0.99	0.35	2.0 × 10 ⁻⁵	3.7 × 10 ⁻⁵

^aOH = ohmic heating.

^bCH = conventional heating.

Table 2

Statistical Parameters Evaluating the Zero- and First-Order Model Performance To Describe Ascorbic Acid Degradation during OH and Conventional Heating of Acerola Serum

process	FQ (Hz)	zero-order model				first-order model			
		R ²	E (%)	χ ²	SEM	R ²	E (%)	χ ²	SEM
OH ^a	10	0.99	4.54	5.2 × 10 ³	9.9 × 10 ³	0.99	0.57	4.5 × 10 ⁻⁵	8.6 × 10 ⁻⁵
OH	10 ²	1.00	4.04	3.6 × 10 ³	6.8 × 10 ³	1.00	0.33	1.9 × 10 ⁻⁵	3.6 × 10 ⁻⁵
OH	10 ³	1.00	5.14	2.2 × 10 ³	4.1 × 10 ³	1.00	0.20	7.1 × 10 ⁻⁶	1.3 × 10 ⁻⁵
OH	10 ⁴	0.99	5.82	2.7 × 10 ³	5.2 × 10 ³	1.00	0.23	8.1 × 10 ⁻⁶	1.5 × 10 ⁻⁵
OH	10 ⁵	0.99	6.55	5.9 × 10 ³	1.1 × 10 ⁴	0.99	0.36	2.3 × 10 ⁻⁵	4.3 × 10 ⁻⁵
CH ^b	0	0.99	6.62	5.9 × 10 ³	1.1 × 10 ⁴	0.99	0.56	4.5 × 10 ⁻⁵	8.5 × 10 ⁻⁵

^a OH = ohmic heating.

^b CH = conventional heating.

Kinetic Parameters of the First-Order Model for Ascorbic Acid Degradation in Acerola Pulp and Acerola Serum during OH and Conventional Heating

Table 3

process	FQ (Hz)	acerola pulp ^d		acerola serum	
		<i>K</i> (min ⁻¹) ^b	<i>D</i> (min) ^b	<i>K</i> (min ⁻¹) ^b	<i>D</i> (min) ^b
OH ^c	10	0.0016 ± 0.0001 a	1480 ± 72 a	0.00151 ± 0.00001 a	1524 ± 9 a
OH	10 ²	0.0012 ± 0.0001 b	1948 ± 50 b	0.00143 ± 0.00008 a	1612 ± 86 a
OH	10 ³	0.0012 ± 0.0001 b	1927 ± 183 b	0.00144 ± 0.00003 a	1599 ± 37 a
OH	10 ⁴	0.0012 ± 0.0001 b	1889 ± 124 b	0.00112 ± 0.00007 b	1923 ± 110 b
OH	10 ⁵	0.0011 ± 0.0001 b	2145 ± 179 b	0.00123 ± 0.00003 b	1867 ± 39 b
CH ^d	0	0.0012 ± 0.0001 b	1860 ± 151 b	0.00118 ± 0.00003 b	1951 ± 56 b

^a Adapted with permission from ref 12 (Copyright 2013 Elsevier, License number 3305350538453).

^b Mean of two replicates ± standard error. Means in the same column with different letters (a and b) are significantly different (*p* < 0.05).

^c OH = ohmic heating.

^d CH = conventional heating.