



Ascorbic acid degradation kinetics of sonicated orange juice during storage and comparison with thermally pasteurised juice

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ABSTRACT

Ascorbic acid degradation kinetics of sonicated orange juice during storage were determined and compared to thermally pasteurised samples. Acoustic energy densities (AED) ranging from 0.30 to 0.81 W/mL and treatment times of 2–10 min were investigated. The degradation kinetics of sonicated samples followed first-order kinetics ($R^2 \geq 0.91$) during processing. During storage ascorbic acid degradation of sonicated samples followed the Weibull model ($R^2 \geq 0.97$) with β values ranging from 0.662 to 0.697. Comparatively, first-order degradation kinetics were observed during storage for thermally pasteurised ($R^2 = 0.98$) and control samples ($R^2 = 0.96$). Increased shelf life based on ascorbic acid retention was found for sonicated samples compared to thermally pasteurised samples. Predicted shelf life for sonicated orange juice ranged from 27 to 33 days compared to 19 days for thermally pasteurised juice during storage at 10 °C. These results indicate that sonication results in enhanced retention of ascorbic acid in orange juice during storage compared to thermal processing.

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1. Introduction

Orange juice is the predominant juice processed by the beverage industry worldwide. Citrus juices are the most popular fruit juices, accounting for greater than 50% of juice in international commerce (Varnam & Sutherland, 1999). Over the past few years while the consumption of fresh citrus fruits has decreased in developed countries, processed fruit juice consumption has increased (Ros-Chumillas, Belissario, Iguaz, & López, 2007). Although thermal processing remains the most widely employed pasteurisation technique, there is growing interest in the development of alternative preservation techniques that minimise changes of organoleptic and nutritional properties.

Power ultrasound has been recognised as a promising processing alternative to conventional thermal treatment in the food industry. Power ultrasound has been reported to be effective against foodborne pathogens found in orange juice (Valero et al., 2007), guava juice (Cheng, Soh, Liew, & The, 2007), apple cider and milk (Dennis, Todd, Wu, & Mingruo, 2006). The propagation of power ultrasound in a liquid induces bubble cavitation due to pressure changes. These resultant micro bubbles collapse violently

in the succeeding compression cycles of propagated ultrasonic waves (Tiwari, Muthukumarappan, O'Donnell, & Cullen, 2008a). This results in a localised high temperatures up to 5000 K, pressures up to 50,000 kPa, and high shearing effects. Consequently, the intense local energy and high pressure bring about a localised pasteurisation effect without causing a significant rise in macro-temperature.

The nutritional quality of orange juice is primarily related to the ascorbic acid content (Zerdin, Rooney, & Vermuë, 2003). Ascorbic acid is thermolabile and highly sensitive to various processing conditions. The mechanism of vitamin C degradation follows aerobic and/or anaerobic pathways and depends upon several processing conditions (Tannenbaum, 1976; Vieira, Teixeira, & Silva, 2000).

Several studies have shown that non-thermal process technologies including high pressure and pulsed electric fields retain a higher level of ascorbic acid relative to thermally processed juices (Cheng et al., 2007; Torregrosa, Esteve, Frigola, & Cortes, 2006; Yeom, Streaker, Zhang, & Min, 2000). However, no studies to date are reported in the literature on the effect of power ultrasound parameters on orange juice ascorbic acid content during storage. The aims of this study were to (i) determine the effect of sonication on the ascorbic acid content of freshly squeezed orange juice and (ii) compare the ascorbic acid degradation kinetics of sonicated and thermally pasteurised during storage at 10 °C.

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2. Materials and methods

2.1. Preparation of orange juice samples

Oranges (*Citrus sinensis* cv. *valencia*) were purchased from a local fruit supplier (Reilly Wholesale Ltd., Dublin, Ireland). Fresh juice was squeezed using a household table top citrus juice extractor (BRAUN GmbH, Kronberg, Germany) and filtered using a double layer cheese cloth to remove pulp.

2.2. Ultrasonic processing

Samples of 80 mL were processed at a constant frequency of 20 kHz using a 1500 W ultrasonic processor (VC 1500, Sonics and Materials Inc., Newtown, USA) with a 19 mm diameter probe. A range of acoustic energy density (AED) values (0.30, 0.33, 0.36, 0.42, 0.47, 0.53, 0.61 and 0.81 W/mL) and treatment times (2, 4, 6, 8 and 10 min) were studied with pulse durations of 5 s on and 5 s off. Temperature was controlled at 25 ± 1.0 °C using a 100 mL jacketed vessel through which water at a flow rate of 0.5 L/min was circulated. The ultrasound probe was submerged to a depth of 25 mm in the sample. All treatments were carried out in triplicate.

2.3. AED determination

AED values were determined using a calorimetric method (Tiwari, Muthukumarappan, O'Donnell, & Cullen, 2008b). Orange juice samples were sonicated with temperature (T) recorded as a function of time under adiabatic conditions using a T-type thermocouple (RS-1315, Radionics, Dublin, Ireland). The initial temperature rise (dT/dt) was determined by polynomial curve fitting. Ultrasonic power (P) and AED (W/mL) values were calculated using Eqs. (1) and (2);

$$P = mC_p(dT/dt)_{t=0} \quad (1)$$

$$\text{AED} = \frac{P}{V} \quad (2)$$

where (dT/dt) is the change in temperature over time ($^{\circ}\text{C s}^{-1}$), C_p is the specific heat of orange juice ($3.73 \text{ kJ kg}^{-1} \text{ }^{\circ}\text{C}^{-1}$), P is ultrasonic power (W), m is sample mass (kg) and V is sample volume (mL).

2.4. Thermal processing

Samples were thermally processed at 98 °C for a holding time of 21 s in a pilot scale tubular heat exchanger (Armfield FT74, HTST/UHT processing unit, Hampshire, England) and cooled to ambient temperature prior to packaging and storage. These pasteurisation conditions were selected with reference to industrial practice where heating of orange juice at 90–99 °C for 15–30 s is carried out (Braddock, 1999).

2.5. Packaging and storage

A total of 50 mL of thermally processed, sonicated and control samples were stored in 100 mL sterilised polypropylene tubes (Fluka, Sigma-Aldrich, Dublin, Ireland) under refrigerated storage conditions of 10 °C (Torregrosa et al., 2006) for 30 days. Samples sonicated at 0.33, 0.47 and 0.81 W/mL at processing times of 2, 6 and 10 min were selected for shelf life studies. These conditions were selected based on preliminary microbial inactivation studies where 5 log reductions were obtained in orange juice. Analysis of samples was carried out at 7, 15, 22 and 30 days storage.

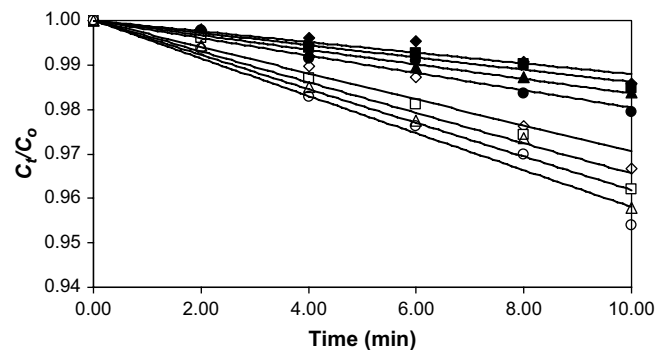


Fig. 1. Ascorbic acid (mg/100 mL) degradation kinetics of sonicated orange juice at AED values of 0.30 (◆), 0.33 (■), 0.36 (▲), 0.42 (●), 0.47 (◇), 0.53 (□), 0.61 (△), and 0.81 (○) W/mL.

2.6. Ascorbic acid determination

Ascorbic acid content was determined following the HPLC (Simadzu Model no: SPD - M10AVP, Simadzu Co., Japan) analytical procedure outlined by Lee and Coates (1999). A total of 10 μL aliquot samples were injected into a Simadzu C18 (15 cm \times 4.6 cm, pore size 5 μm) coupled with HyperODS guard column; 25 mL juice samples were pipetted into 50 mL centrifuge tubes containing 5 mL of 2.5% metaphosphoric acid. Samples were centrifuged (Sanyo MSE Mistral 3000i, UK) for 10 min at 2000 \times g and 4 °C; 5 mL of the supernatant was filtered through 0.45 μm PTFE syringe filters (Phenomenex, U.K) and placed in an autosampler vial. The mobile phase was 25 mM KH_2PO_4 (adjusted to pH 3.0 with phosphoric acid) with a flow rate of 1 mL/min. Eluate was monitored by UV detection at 245 nm. Chromatograms were recorded and processed with EZStart Chromatography Software V.7.2. 1. Results were reported as mg/100 mL of orange juice.

2.7. Ascorbic acid degradation kinetics

Ascorbic acid degradation was modelled using first order and Weibull models (Eqs. (3) and (4)). The Weibull model is flexible owing to the inclusion of a shape constant in addition to the rate constant and has been employed to describe microbial, enzymatic and chemical degradation kinetics (Cunha, Oliveira, & Oliveira, 1998; Manso, Oliveira, Oliveira, & Frias, 2001).

$$C_t = C_0 \times e^{-K_{aa}t} \quad (3)$$

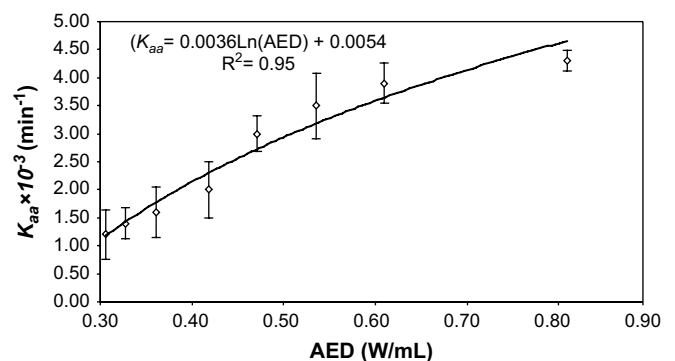


Fig. 2. Changes in ascorbic acid rate constant $K_{aa} \times 10^{-3}$ (min) as a function of AED (W/mL).

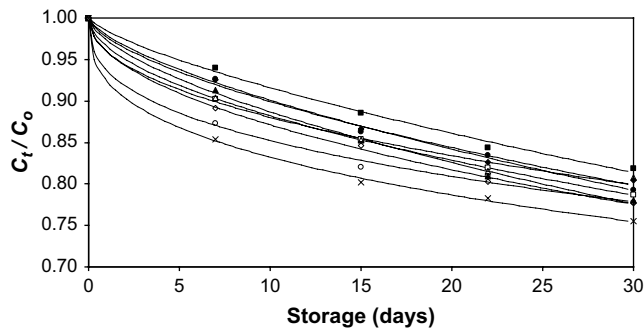


Fig. 3. Ascorbic acid (mg/100 mL) degradation kinetics of sonicated orange juice during storage at 10 °C for 2 min at (■) 0.33, (◆) 0.47, (▲) 0.81; for 6 min at (●) 0.33, (□) 0.47, (◇) 0.81, and 10 min at (△) 0.47, (○) 0.47, and (×) 0.81 WmL⁻¹, respectively.

$$C_t = C_0 \times e^{-(K_{aa}t)^\beta} \quad (4)$$

where C_t is the ascorbic acid concentration at a time t , C_0 is the initial ascorbic acid concentration, K_{aa} is the rate constant and β (dimensionless) is the shape constant. Modelling and analysis of variance were performed using TableCurve™ 2D v5.01 (SYSTAT Software Inc., Chicago, USA).

3. Results and discussion

3.1. Effect of AED on ascorbic acid

A significant ($p < 0.05$) decrease in orange juice ascorbic acid content (mg/100 mL) was observed as a function of AED and treatment time. However at the highest AED value (0.81 W/mL) and treatment time (10 min), the largest reduction found in ascorbic acid after sonication was less than 5%. Ascorbic acid degradation was found to follow first-order kinetics ($R^2 > 0.91$, $SE < 0.1$) (Fig. 1). The ascorbic acid degradation rate constant (K_{aa}) increased from $1.2 \times 10^{-3} \text{ min}^{-1}$ ($R^2 = 0.91$, $SE = 0.09$) to $4.3 \times 10^{-3} \text{ min}^{-1}$ ($R^2 = 0.97$, $SE = 0.04$) with an increase in AED from 0.30 to 0.81 W/mL. The reaction rate constants were found to increase logarithmically with AED ($R^2 = 0.95$) (Fig. 2). The rate constants obtained are lower than those reported for thermally processed orange juice (Polydera, Stoforos, & Taoukis, 2003), indicating improved stability of ascorbic acid in sonicated orange juice compared to thermally processed samples.

Degradation of ascorbic acid is reported to follow first-order kinetics. The degradation curve divides into two linear sections corresponding to aerobic and anaerobic degradation (Ariahu, Adekunle, & Nkpa, 1997; Blasco, Esteve, Frigola, & Rodrigo, 2004; Eison-Perchonok & Downes, 1982; Kennedy, Rivera, Lloyd, Warner, & Jumel, 1992; Nagy, 1980; Robertson & Saminego-Esguerra, 1986). Sonication results in a reduction of dissolved oxygen, a critical parameter influencing the stability of ascorbic acid (Solomon & Svanberg, 1995). Ascorbic acid degradation during sonication may

Table 2

The effect of AED and processing time on Weibull model shape factors (β)

Processing time (min)	0.33 W/mL	0.47 W/mL	0.81 W/mL
2	0.662	0.671	0.680
6	0.672	0.680	0.684
10	0.677	0.688	0.697

be due to free radical formation (Portenlänger & Heusinger, 1992). Hydroxyl radical formation is found to increase with degassing. Sonication cavities can be filled with water vapour and gases dissolved in the juice, such as O_2 and N_2 (Korn, Prim, & deSousa, 2002). The interactions between free radicals and ascorbic acid may occur at the gas–liquid interfaces. In summary, ascorbic acid degradation may follow one or both of the following pathways:

Ascorbic acid \rightarrow thermolysis (inside bubbles) and triggering of Maillard reaction

Ascorbic acid \rightarrow reaction with OH^- \rightarrow HC–OH and production of oxidative products on the surface of bubbles

Thereby, sonication can be related to advanced oxidative processes since both pathways are associated with the production and use of hydroxyl radicals (Hart & Henglein, 1985; Petrier et al., 2007).

3.2. Storage studies

During refrigerated storage at 10 °C, ascorbic acid content decreased with storage time for control, sonicated and thermally processed samples as shown in Table 1. Ascorbic acid degradation in sonicated samples followed the Weibull model (Eq. (4)) with $R^2 > 0.97$ (Fig. 3). The Weibull model shape factors listed in Table 2, indicate that the ascorbic acid degradation curves were concave upward ($\beta < 1$), which can also be seen from Fig. 3. Upward concavity ($\beta < 1$) as observed in this study indicates an increased stability of ascorbic acid during storage, whereas downward concavity ($\beta > 1$) would indicate higher degradation rates. The β values were found to correlate linearly with treatment time and AED (Fig. 4a, b).

The degradation of ascorbic acid for thermally processed and control samples followed first-order kinetics during storage (Fig. 5). First-order kinetics equates to the Weibull model with $\beta = 1$. Storage rate constants (K_{saa}) for thermally processed and control juice samples were $4.44 \times 10^{-2} \text{ days}^{-1}$ ($R^2 = 0.96$) and $2.19 \times 10^{-2} \text{ days}^{-1}$ ($R^2 = 0.98$). K_{saa} for thermally processed juice was slightly higher than previously reported values ($2.31 \times 10^{-2} \text{ days}^{-1}$) for thermally processed reconstituted orange juice processed at 80 °C for 30 s during storage at 10 °C (Polydera et al., 2003). This difference may be caused by the higher processing temperature (98 °C) employed in our study.

Ascorbic acid is a heat sensitive nutrient (Saguy, Kopelman, & Mizrah, 1978). High processing temperatures and storage cause a loss of ascorbic acid (Nagy & Smoot, 1977). Thermally processed orange juice had a significantly lower concentration of ascorbic acid

Table 1

Changes in ascorbic acid of sonicated, thermally processed and control (untreated) samples

Storage time (days)	Sonicated orange juice ^a			Control	Thermally processed
	0.33 W/mL	0.47 W/mL	0.88 W/mL		
0	45.94 ^b	45.12 ^b	44.21 ^b	46.62 ^b	43.29 ^b
7	41.25 ^c	39.83 ^c	38.97 ^c	39.79 ^c	32.08 ^c
15	38.93 ^d	37.44 ^d	36.58 ^d	33.04 ^d	24.36 ^d
22	37.19 ^e	36.93 ^d	35.71 ^e	25.96 ^e	18.67 ^e
30	36.86 ^e	35.49 ^e	34.44 ^f	24.91 ^f	11.22 ^f

^{bcd} Values followed by same letter within a row are not significant ($p < 0.05$).

^a Samples processed at treatment time of 10 min.

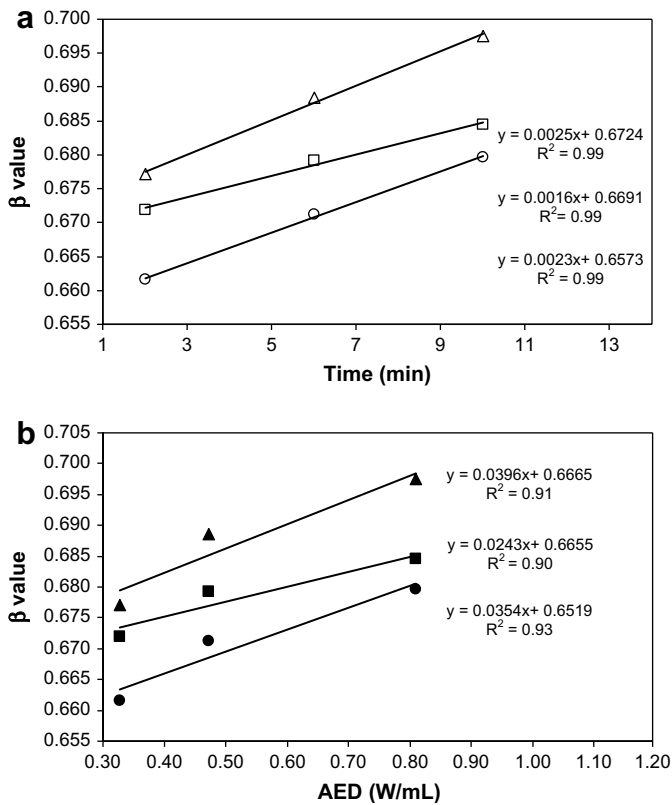


Fig. 4. Change in Weibull model shape factor β (dimensionless) with respect to (a) processing time at varying AED level of (○) 0.33, (□) 0.47, and (△) 0.81 W/mL and (b) AED level at varying time of (●) 2, (■) 6, and (▲) 10 min, respectively.

than sonicated and control orange juice during storage at 10 °C ($p < 0.05$) due to the higher processing temperature. Kabasakalis, Siopidou, and Moshatou (2000) reported a 60–67% loss of ascorbic acid content in commercially processed orange juice and a 7–13% loss in fresh unprocessed orange juice during a refrigerated storage period of 31 days.

Ascorbic acid storage degradation rate constants (K_{saa}) for sonicated orange juice samples determined by Eq. (4) were found to increase exponentially with AED (Fig. 6). The reaction mechanism of ascorbic acid decomposition in foods has been extensively studied (Liao & Seib, 1988). It has been reported that ascorbic acid degrades aerobically and anaerobically during storage, at rates depending on storage conditions, packaging and the processing method employed (Kabasakalis et al., 2000; Kennedy et al., 1992).

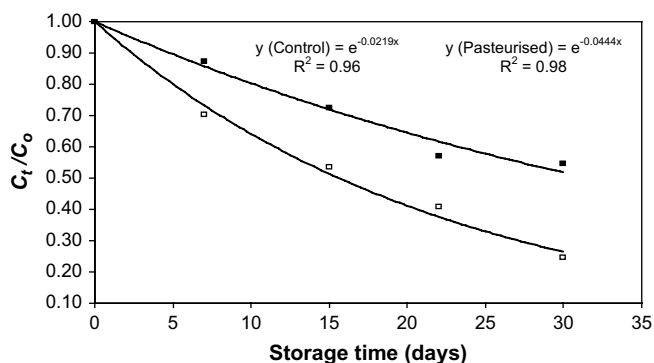


Fig. 5. Ascorbic acid (mg/100 mL) degradation kinetics of thermally processed (■) and control (□) orange juice during storage at 10 °C.

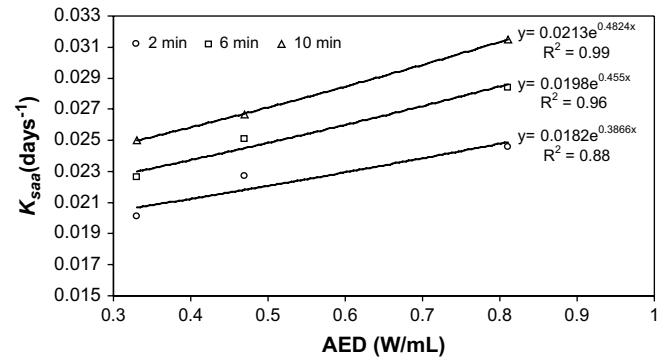


Fig. 6. Change in storage rate constant (K_{saa} , days⁻¹) with respect to AED (W/mL⁻¹)

Table 3

Predicted shelf life of control, thermally processed and sonicated orange juice samples

Treatment condition	Shelf life (days)	
	USRDA	EU
Control	27	38
Thermally processed	13	19
AED 0.33 W/mL; 6 min	27	33
AED 0.47 W/mL; 6 min	24	30
AED 0.81 W/mL; 6 min	21	27

3.3. Ascorbic acid shelf life prediction

Orange juice shelf life was estimated in accordance with relevant EU and US guidelines. In the EU the ascorbic acid content of orange juice should be higher than 20 mg/100 mL at the expiration date (Polydera et al., 2003). The U.S. recommended daily allowances (USRDA) state that the concentration of ascorbic acid in orange juice should be at least 25 mg/100 mL at the expiration date (Yeom et al., 2000).

The shelf life (SL) of sonicated, thermally processed and control juices during storage at 10 °C was calculated by extrapolating Eqs. (3) and (4). Shelf life of thermally processed juice was found to be 13 and 19 days for US and EU guidelines, respectively (Table 3). Shelf life of sonicated juice was found to be significantly ($p < 0.05$) higher than thermally processed juice. The lower degradation rate found in sonicated juices results in a longer shelf life than that for thermally processed juice. The relative decrease in the ascorbic acid degradation rate at other storage temperatures might be more or less pronounced than the observed at 10 °C.

4. Conclusions

A significant decrease in orange juice ascorbic acid content was observed as a function of AED and treatment time. However at the highest AED value (0.81 W/mL) and treatment time (10 min), the largest reduction found in ascorbic acid after sonication was less than 5%. During storage at 10 °C sonicated juice was found to have higher retention of ascorbic acid compared to thermally processed and control samples. This work demonstrates that sonication results in improved retention of ascorbic acid in orange juice compared to thermal processing.

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