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Innovative Food Science & Emerging Technologies

Innovative Food Science and Emerging Technologies 6 (2005) 1-9

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Quality degradation kinetics of pasteurised and high pressure processed fresh Navel orange juice: Nutritional parameters and shelf life

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Received 29 May 2004; accepted 25 October 2004

Abstract

A kinetic study of post processing quality loss was conducted after high pressure processing (600 MPa, 40 °C, 4 min) or thermal pasteurisation (80 °C, 60 s) of fresh Navel orange juice. Selection of processing conditions was mainly based on pectin methylesterase inactivation. Ascorbic acid loss, colour, viscosity and sensory characteristics were measured during storage at different isothermal conditions (0–30 °C). Increased shelf life (based on ascorbic acid retention) was achieved for high pressurised compared to thermally pasteurised juice, ranging from 49% (storage at 15 °C) to 112% (storage at 0 °C). Activation energy values for ascorbic acid loss were 68.5 and 53.1 kJ/mol, respectively, for high pressurised and thermally treated juice. High pressure processing resulted in better retention of flavour of untreated juice and superior sensory characteristics compared to thermal pasteurisation. Colour change was linearly correlated to ascorbic acid loss for both types of processing. Slightly higher apparent viscosity values were determined for high pressurised juice. © 2004 Elsevier Ltd. All rights reserved.

Keywords: Kinetics; Shelf life; Ascorbic acid; High pressure; Pasteurisation; Orange juice

Industrial relevance: Application of high hydrostatic pressure on orange juice industry. Fresh orange juice is a product of high commercial and nutritional value due to its rich vitamin C content and its desired sensory characteristics. High Hydrostatic Pressure (HHP) is an alternative non-thermal technology that has been proposed for application on orange juice. Such a treatment denaturates enzymes and eliminates microorganisms responsible for spoilage of orange juice without detrimental effects on the sensory and nutritional quality of juice. The effect of HHP on the stability of fresh orange juice has been studied by different research groups, while orange juices processed with the new technology have already been commercially available in Japan, U.S.A., Mexico and Europe. However, a systematic kinetic approach of the effect of HHP on different quality indexes (not only microbial spoilage) immediately after processing, as well as during a long term storage of the processed orange juice is needed, in order to achieve an optimal process design and a successful application of the new technology in orange juice industry. Such kinetic data for parameters related to the quality and nutritional value of fresh orange juice were gathered in the present work providing therefore industry with useful information for the HHP stabilization of orange juice and the production of a high quality product. Due to the great benefits of HHP compared to the conventional pasteurization that emerged from this work regarding the quality, shelf life and nutritional characteristics of fresh orange juice.

1. Introduction

High hydrostatic pressure (HHP) processing has been introduced as an alternative non-thermal technology for

preservation of orange juice. A major problem associated with orange juice quality deterioration during storage is cloud loss accompanied by gelation of juice concentrates. This action has been primarily attributed to the activity of pectin methylesterase (PME) (Cameron, Baker, & Grohmann, 1997; 1998; Versteeg, Rombouts, Spaansen, & Pilnik, 1980), the inactivation of which is generally used to determine the intensity of thermal processing during

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^{1466-8564/\$ -} see front matter ${\odot}$ 2004 Elsevier Ltd. All rights reserved. doi:10.1016/j.ifset.2004.10.004

commercial pasteurisation (Snir, Koehler, Sims, & Wicker, 1996; Versteeg et al., 1980). Pasteurisation conditions used to inactivate the most heat resistant PME iso-enzyme (90 °C for 1 min) reduce "freshness", affecting sensory and nutritional characteristics of orange juice (Manso, Oliveira, Oliveira, & Frias, 2001; Yeom, Streaker, Zhang, & Min, 2000). HHP achieves inactivation of microorganisms (Linton, McClements, & Patterson, 1999; Parish, 1998a; Zook, Parish, Braddock, & Balaban, 1999), inactivation of orange PME (Basak, Ramaswamy, & Simpson, 2001; Nienaber & Shellhammer, 2001a; Parish, 1998a; Van den Broeck, Ludikhuyze, Van Loey, & Hendrickx, 2000) and denaturation of several other enzymes (Cano, Hernández, & De Ancos, 1997; Weemaes, Ludikhuyze, Van den Broeck, & Hendrickx, 1999), while minimally affecting quality and organoleptic characteristics (Fernández-García, Butz, Bognar, & Tauscher, 2001; Nienaber & Shellhammer, 2001b). PME exhibits greater heat and pressure resistance compared to that of common spoilage microorganisms of orange juice and thus can be used as processing index for both HHP and thermal process (Goodner, Braddock, & Parish, 1998; Versteeg et al., 1980).

The effect of HHP on post processing quality loss of orange juice is an important issue for study. During storage, orange juice undergoes a number of deteriorative reactions resulting in quality degradation of the product. Among them, ascorbic acid is degraded, following two consecutive or parallel pathways, aerobically and anaerobically, at rates depending on storage conditions, packaging and type of processing (Kennedy, Rivera, Lloyd, Warner, & Jumel, 1992; Sadler, Parish, Van Clief, & Davis, 1997; Tawfik & Huyghebaert, 1998). Remaining ascorbic acid constitutes an important quality indicator often defining the shelf life of the product (Lee & Chen, 1998; Lee & Coates, 1999). Loss of nutrients other than ascorbic acid, cloud loss, microbial spoilage, development of off-flavour, changes in colour, texture and appearance are other phenomena that influence the overall quality of orange juice (Goyle & Ojha, 1998; Roig, Bello, Rivera, & Kennedy, 1999).

Application of HHP can lead to an extended shelf life of orange juice compared to that of untreated juice with minimal product quality loss and a good retention of fresh-like flavour (Donsi, Ferrari, & Di Matteo, 1996; Nienaber & Shellhammer, 2001b). Although several studies reported retention of the overall quality of high pressure processed orange juice and increase of its shelf life compared to that of untreated juice, few works compare the effect of an alternative HHP process with that of a conventional heat pasteurisation on orange juice quality parameters during storage, studying only specific quality indicators, e.g. sensory characteristics or microbial growth (Parish, 1998b). A comparative study of HHP and thermal pasteurisation concerning their effect on a number of qualitative characteristics during storage has been reported for reconstituted from frozen concentrate orange juice (Polydera, Stoforos, & Taoukis, 2003), which is the most

common type of orange juice produced worldwide. However, the increasing consumers' demand for minimally processed products and the main advantage of HHP to retain the sensory and quality characteristics of the processed product make fresh orange juice the most likely type of juice for HHP application.

The objective of this work was to comparatively evaluate the effect of conventional thermal pasteurisation and alternative HHP processing on post processing quality loss of fresh Navel orange juice. Selection of process conditions was based on PME inactivation kinetics. Additional criteria considered were sensory quality and microbial stability of orange juice. The shelf life of juices was determined at different storage temperatures within the broad range of 0–30 °C. Determination of shelf life was mainly based on ascorbic acid loss kinetics, while a variety of other quality parameters such as sensory characteristics, colour and viscosity were also investigated.

2. Materials and methods

2.1. Juice samples

Orange juice of greek Navel variety (*Citrus sinensis*), from a Food Manufacturing Coalition (FMC) production line of a commercial juice plant in Southern Greece was obtained. No thermal treatment was applied. The juice was immediately frozen in a forced circulation freezer (MDF-U442, SANYO Electric, Japan) and kept at -40 °C until use.

2.2. High pressure processing

High pressure treatments were achieved using a laboratory pilot scale HHP equipment with a maximum operating pressure of 1000 MPa (Food Pressure Unit FPU 1.01, Resato International, Roden, Holland) consisting of an operation high pressure unit with a pressure intensifier, a high pressure vessel of 1.5 L in volume and a multivessel system consisting of six vessels of 45 mL capacity each. All high pressure vessels were surrounded by a liquid circulating jacket connected to a heating–cooling system. The pressure transmitting fluid used was polyglycol ISO viscosity class VG 15 (Resato International).

For the HHP experiments, polypropylene bottles of 150 mL capacity with screw-cup closures were used. Fifty bottles were filled with orange juice and placed into the large vessel for processing. The desired value of pressure (600 MPa) was set and after pressure build-up (about 1 min), the pressure vessel was isolated. This point defined the time zero of the process. Pressure was released after a preset time interval (4 min) by opening the pressure valve. The initial temperature increase during pressure build-up (about 3 °C/100 MPa) was taken into consideration in order to achieve an average operating temperature of 40 °C during pressurisation.



Fig. 1. Ascorbic acid loss during storage of (a) high pressurised and (b) thermally pasteurised orange juice at 0-30 °C.

Pressure and temperature were constantly monitored and recorded (in 1 s intervals) during the process.

2.3. Thermal pasteurisation

Orange juice was pasteurised in a pilot scale pasteuriser with a tubular heat exchanger (Armfield FT74, HTST/UHT Processing Unit, Hampshire, England) at 80 °C for 60 s. The pasteurised juice was aseptically transferred into packages identical to the ones used for high pressure pasteurisation.

2.4. Shelf life study

Samples of thermally and high pressure pasteurised orange juice were stored immediately after processing (time 0) at five different isothermal conditions (0, 5, 10, 15 and 30 °C) in temperature programmable control cabinets (Sanyo MIR 153, Sanyo Electric Co, Ova-Gun, Gunma, Japan). Ten samples per process and storage condition were used. The temperature was constantly recorded by type T thermocouples and a multichanell datalogger (CR10X, Campbell Scientific, Leicestershire, UK). Samples were evaluated at time 0 and at regular time intervals according to the experimental design. Different quality parameters (Lascorbic acid, colour, sensory characteristics, viscosity) were measured.

2.5. Determination of L-ascorbic acid

L-Ascorbic acid concentration was determined using an HPLC method. Samples of 1 mL of orange juice were

extracted with equal volumes of 4.5% (w/v) metaphosphoric acid solution and filtered through a 0.45-µm GHP Acrodisc filter. An aliquot then was injected into the chromatographic column. The chromatographic system (HP 1100 Series, Waldbronn, Germany) consisted of a quaternary pump, a vacuum degasser, a Rheodyne 20 µL injection loop, a Diode-Array Detector, and it was controlled through a HP ChemStation software. A Hypersil ODS column (250×4.6 mm, particle size 5 µm) fitted with a Hypersil ODS guard column was utilised with a mobile phase of HPLC grade water with metaphosphoric acid to pH 2.2 at a flow rate of 0.5 mL/min. The detection was at 245 nm (Oruña-Concha, González-Castro, López-Hernández, & Simal-Lozano, 1998). Results were calculated as mg of L-ascorbic acid per 100 mL of orange juice. Each sample was prepared and analysed in duplicate.

2.6. Sensory evaluation

A panel of six trained panellists was used for sensory evaluation during storage of orange juice. The panellists were selected by standard ISO 3972 (ISO, 1991) procedures and trained in taste recognition with orange juice samples of variable quality levels. Each day of analysis, randomly selected samples of both HHP and thermal treatments were removed from the storage cabinets, tempered to ambient temperature and then sensory evaluated. Scores were assigned for the different flavour characteristics of the orange juice as well for total impression of the juice using a nine grade hedonic scale. A mean value equal to 5 was determined as the acceptance limit signalling the end of the shelf life of juice.

Table 1

10001010 100010000 100010000 100010000 10000000000	Ascorbic acid loss rates, k (davs ^{-1})	¹), during storage of high	pressurised and thermally paster	urised orange juice at 0–30 °C
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Storage temperature (°C)	High pressurized orange juice		Thermally pasteurized orange juice	
	$k \text{ (days}^{-1})$	R^2	$k \text{ (days}^{-1})$	R^2
0	$0.00400 \pm 4.60 \times 10^{-4a}$	0.94	$0.00774 \pm 3.76 \times 10^{-4}$	0.98
5	$0.00580 \pm 1.51 \times 10^{-4}$	0.99	$0.0108 \pm 1.07 \times 10^{-3}$	0.94
10	$0.00852 \pm 3.13 \times 10^{-4}$	0.99	$0.0176 \pm 1.08 \times 10^{-3}$	0.97
15	$0.0246 \pm 1.19 \times 10^{-3}$	0.99	$0.0343 \pm 9.48 imes 10^{-4}$	0.99
30	$0.0684 \pm 5.75 \times 10^{-3}$	0.95	$0.0715 \pm 6.86 \times 10^{-3}$	0.97

^a Standard error.



Fig. 2. Effect of storage temperature $(0-30 \ ^{\circ}C)$ on ascorbic acid loss rate of high pressurised and thermally pasteurised orange juice.

2.7. Colour measurement

Colour was measured using a CR-200 Minolta Chroma meter (Minolta, Chuo-Ku, Osaka, Japan) with an 8 mm measuring area. A Minolta standard-white reflector plate was used to standardise the instrument under CIE (Commission International de L'Eclairage) illuminant C conditions. Samples of orange juice were filled into 25 mm glass petri dishes and CIELab values were determined. All samples were analysed in duplicate.

2.8. Viscosity

Viscosity of orange juice was measured using a computer controlled rotary viscometer RHEOTEST RC1 (Medingen, Radeburg, Germany) consisting of an electronic unit with standard DIN coaxial cylinder measuring systems of different viscosity ranges, a temperature measuring sensor Pt100, a thermostatting device FTK-CC (-10 to 90 °C) and a RHEO 2000 software. The measuring system used was a double gap cylinder DG DIN with a viscosity range from 0.001 to 1.30 Pa ·s. Shear stress and viscosity were measured and recorded at different shear rates in the range from 4 to 500 s⁻¹. All measurements were conducted at room temperature (25 °C).

3. Results and discussion

3.1. Selection of processing conditions

Thermal pasteurisation conditions (80 °C, 60 s) were chosen among different conditions used in industrial practice taking also into consideration PME thermal inactivation kinetics (Polydera, Galanou, Stoforos, & Taoukis, 2004). About 95% inactivation of initial PME activity of untreated juice was achieved. The remaining 5% of the enzyme activity corresponds to the more heat resistant isoenzyme which can cause cloud loss after long periods of storage (Cameron et al., 1997; Versteeg et al., 1980). More intense thermal treatment in order to inactivate this portion would greatly affect sensory characteristics of orange juice without contributing substantially to further PME inactivation.

Selection of HHP processing conditions was mainly based on PME inactivation kinetics (Polydera et al., 2004). From the conditions providing adequate PME inactivation, the ones resulting in optimum sensory quality were selected. These conditions also exceeded process requirements for microbial stability of orange juice (Polydera et al., 2003). A treatment of fresh greek Navel orange juice at 600 MPa and 40 °C for 4 min can cause inactivation of the sensitive isoenzyme, leading to a remaining PME activity equal to approximately 7% of the initial activity of untreated juice (similar to that after thermal pasteurisation). Cloud stabilization of HHP processed orange juice has been reported for values up to 20% of remaining enzymatic activity (Boff, Truong, Min, & Shellhammer, 2003; Goodner et al., 1998; Nienaber & Shellhammer, 2001b) during storage at 4-30 °C for long time periods (4 months). In comparison, such inactivation values for a thermally processed orange juice would lead to cloud loss within 4 weeks (Cameron et al., 1997; Versteeg et al., 1980). Application of more intense HHP conditions (pressure, temperature or time) resulted in faster or further inactivation of PME, affecting however negatively the sensory quality of orange juice. The effect on the organoleptic characteristics was comparatively evaluated through sensory testing of orange juice after treatment at various combinations of pressure (400-900 MPa), temperature (30-60 °C) and time (2-10 min), leading to

Table 2

Shelf life (days) of high pressure or thermally pasteurized orange juice stored at 0, 5, 10, 15 and 30 $^\circ C$

Shelf life (days)					
Storage temperature (°C)	Based on 50% ascorbic	acid loss	Based on sensory evaluation		
	High pressurized orange juice	Thermally pasteurized orange juice	High pressurized orange juice	Thermally pasteurized orange juice	
0	187	88	147	111	
5	109	58	89	66	
10	64	39	55	40	
15	39	26	35	24	
30	9	9	10	6	



Fig. 3. Change of total sensory score during storage of (a) high pressurised and (b) thermally pasteurised orange juice at 0-30 °C.

inactivation of the labile portion of the enzyme. The selected processing conditions of 600 MPa and 40 °C for 4 min also did not affect nutritional parameters of orange juice like ascorbic acid concentration or total antioxidant activity (Polydera, Stoforos, & Taoukis, in press).

After processing, thermal or HHP, microbiological measurements confirmed that microorganisms responsible for orange juice spoilage had been inactivated after both treatments (no microbial growth was observed for all samples tested).

3.2. Ascorbic acid loss kinetics-shelf life determination

Ascorbic acid loss was found to follow apparent first order kinetics (Eq. (1)) during storage of both high pressure and thermally pasteurised orange juice as depicted in Fig. 1a and b, respectively. Symbols refer to the average of experimental values, while lines correspond to the fitted values of Eq. (1) to experimental data.

$$C = C_0 \cdot \exp(-k \cdot t) \tag{1}$$

where *C*: ascorbic acid (AA) concentration (mg AA/100 mL juice) at time *t*, C_0 : ascorbic acid concentration at time 0, *k*: ascorbic acid loss rate (days⁻¹), *t*: storage time (days).

Ascorbic acid loss rates as determined by Eq. (1) and experimental data through a least square fitting procedure are shown in Table 1 (together with the R^2 of the correlation). Lower ascorbic acid loss rates were found in the case of high pressurised orange juice compared to the respective values of conventionally pasteurised juice at all storage temperatures used. The retention of ascorbic acid after storage of high pressurised orange juice for 1 month at 5 °C was 84%, in contrast to thermal treatment that led to a retention of ascorbic acid equal to 72%. A possible explanation for the lower ascorbic acid degradation rates during storage of high pressure treated orange juice could be a loss of availability of metal ions (e.g. iron, copper), catalysing the ascorbic acid degradation, due to their hydration or the formation of complexes with chelating agents, such as citric acid, reported to be favoured by high pressure (Cheah & Ledward, 1995, 1997). The destruction of peroxides by high pressure application (Cheah & Ledward, 1995) may also be a possible reason for the retardation of ascorbic acid degradation after HHP treatment of orange juice. The same trend was observed for Valencia orange juice reconstituted from frozen concentrate measured in a previous study (Polydera et al., 2003). It is also worth noting that the respective rates of ascorbic acid degradation in the reconstituted juice, at chill chain temperatures (0-10)°C) were significantly higher than the ones in the fresh juice for both high pressure and thermal treatments.

The effect of storage temperature on ascorbic acid degradation rate was described adequately by Arrhenius kinetics (Eq. (2)), as illustrated in Fig. 2 for both high pressure and thermally treated orange juice.

$$k_T = k_{\rm ref} \cdot \exp\left[-\frac{E_{\rm A}}{R}\left(\frac{1}{T} - \frac{1}{T_{\rm ref}}\right)\right]$$
(2)

where k_T : ascorbic acid loss rate at a storage temperature T, k_{ref} : ascorbic acid loss rate at a reference temperature T_{ref} , E_A : activation energy (J/mol), R: gas constant (8.314 J/ (mol \cdot K)) and temperatures in absolute scale (K).

The activation energy was determined to be 68.5 kJ/mol (R^2 =0.97) and 53.1 kJ/mol (R^2 =0.97) for high pressurised and thermally pasteurised orange juice, respectively, suggesting greater temperature dependence of the

Table 3

Sensory loss rates, k (days⁻¹), during storage of high pressurised and thermally pasteurised orange juice at 0–30 °C

Storage temperature (°C)	High pressurized orange juice		Thermally pasteurized orange juice	
	$k \text{ (days}^{-1}\text{)}$	R^2	$k \text{ (days}^{-1})$	R^2
0	$0.0282 \pm 1.51 \times 10^{-3a}$	0.99	$0.0299 \pm 2.93 \times 10^{-3}$	0.93
5	$0.0408 \pm 2.47 imes 10^{-3}$	0.98	$0.0459 \pm 2.79 \times 10^{-3}$	0.97
10	$0.0583 \pm 6.32 \times 10^{-3}$	0.91	$0.0630 \pm 4.40 \times 10^{-3}$	0.91
15	$0.102 \pm 1.46 \times 10^{-2}$	0.89	$0.126 \pm 8.80 \times 10^{-3}$	0.91
30	$0.413 \pm 5.51 \times 10^{-2}$	0.95	$0.511 \pm 10 \times 10^{-2}$	0.87

^a Standard error.



Fig. 4. Colour change during storage of (a) high pressurised and (b) thermally pasteurised orange juice at 0-30 °C.

ascorbic acid degradation rate for high pressurised orange juice.

The decrease of ascorbic acid concentration to levels unacceptable by legislation or industrial practice often defines orange juice shelf life. In the present work, shelf life of orange juice was estimated as the time period in which there is a 50% ascorbic acid loss. The shelf life (t) of high pressure and heat pasteurised juice at different storage temperatures was therefore calculated through Eq. (1), substituting C with 0.5 C_0 and k with the predicted value from Arrhenius equation (Eq. (2)) for each storage temperature (Table 1). The slower ascorbic acid loss rates during storage of high pressurised orange juice led to a significant extension of its shelf life compared to that of the conventionally pasteurised juice (Table 2). The shelf life increase of high pressure processed compared to pasteurised juice ranged from 13 days (49% increase) for storage at 15 °C to 99 days (112% increase) for storage at 0 °C. When stored at 30 °C, similar shelf life values were found for juices of both treatments. The above determined shelf life values for fresh orange juice (Table 2) were up to double the respective values reported by Polydera et al. (2003) for reconstituted Valencia orange juice.

3.3. Sensory evaluation

According to the sensory evaluation that was conducted, the characteristic flavour and aroma of untreated fresh orange juice were retained immediately after HHP processing, while conventionally pasteurised orange juice led to distinguishable differences. Moreover, the sensory quality of high pressure treated juice was judged superior than the pasteurised one during storage at all temperature conditions tested. Total sensory score (in the 9-point hedonic scale) as a function of storage time at 0-30 °C is depicted in Fig. 3 for both high pressure treated and pasteurised juices. Symbols refer to experimental values, while lines represent the fitted values of a zero order equation to experimental data. The respective shelf life values, as they were determined based on sensory evaluation, are illustrated in Table 2. A mean value equal to 5 was judged as the acceptance limit to determine the end of the shelf life of juice. Similarly to the results of shelf life determination based on ascorbic acid loss kinetics, lower shelf life values were found for conventionally pasteurised compared to high pressurised orange juice according to sensory evaluation. It is worth noting that HHP substantially delays both nutrient and sensory quality loss as compared to conventional thermal processing (Tables 1 and 3, respectively). However, the difference in sensory loss rates is less pronounced than the respective difference in nutrient loss rates.

3.4. Change of colour during storage of orange juice

The colour of both high pressure and thermally pasteurised orange juice was estimated as a function of storage time at 0-30 °C using Eq. (3) and illustrated in Fig. 4a and b, respectively.

$$C = \sqrt{a^2 + b^2} \tag{3}$$

where C: chroma, a: redness and b: yellowness.

Browning of orange juices was observed at all storage conditions tested. Similarly to ascorbic acid loss, which is

Table 4

Browning rates, k (days⁻¹), during storage of high pressurised and thermally pasteurised orange juice at 0–30 $^{\circ}$ C

Storage temperature (°C)	High pressurized orange juice		Thermally pasteurized orange juice	
	$k (\text{days}^{-1})$	R^2	$k \text{ (days}^{-1})$	R^2
0	$0.000652 \pm 1.25 \times 10^{-4a}$	0.86	$0.00160 \pm 2.93 \times 10^{-4}$	0.75
5	$0.00166 \pm 1.94 \times 10^{-4}$	0.84	$0.00210\pm2.61 imes10^{-4}$	0.91
10	$0.00208 \pm 2.42 \times 10^{-4}$	0.91	$0.00404 \pm 2.05 \times 10^{-4}$	0.99
15	$0.00618 \pm 6.84 \times 10^{-4}$	0.94	$0.00876 \pm 1.50 \times 10^{-3}$	0.88
30	$0.0219 \pm 1.40 imes 10^{-3}$	0.99	$0.0210 \pm 1.69 \times 10^{-3}$	0.98

^a Standard error.



Fig. 5. Effect of storage temperature $(0-30 \ ^{\circ}C)$ on browning rate of high pressurised and thermally pasteurised orange juice.

greatly related to browning of orange juice (Roig et al., 1999), the change of colour during storage was found to follow first order kinetics (Eq. (4)) for both types of processing.

$$C = C_0 \cdot \exp(-k \cdot t) \tag{4}$$

where C: colour of orange juice at time t, C_0 : colour at time 0 (after processing), k: rate of colour change (days⁻¹) and t: storage time (days).

Rates of colour change for each storage condition were determined by Eq. (4) and experimental data through a least square fitting procedure and shown in Table 4 (together with the R^2 values of the correlation). HHP treatment led to lower rates of colour change compared to thermal pasteurisation at all storage temperatures studied, except at 30 °C (which is above the range of normal storage). Increase of storage temperature resulted in higher rates of browning of orange juice. This effect of storage temperature on browning rate was described adequately by Arrhenius kinetics (Eq. (2)), as illustrated in Fig. 5 for both high pressure and thermally



Fig. 6. The % colour change as a function of the % ascorbic acid loss for the same temperature and time storage conditions of HHP treated (\bullet) and thermally pasteurised (\bigcirc) orange juice.

Table 5

Consistency index, K (Pa · sⁿ), of high pressurised and thermally pasteurised orange juice during storage at 10 °C

Storage High provide the High provide High provide \overline{K} (Pa · s	High pressurised juice		Thermally pasteurised juice		
	K (Pa · s ⁿ)	R^2	K (Pa · s ⁿ)	R^2	
)	$0.052{\pm}2.03{\times}10^{-3a}$	0.94	$0.036 \pm 1.83 \times 10^{-3}$	0.92	
7	$0.039 \pm 1.31 \times 10^{-3}$	0.85	$0.023 \pm 3.87 \times 10^{-4}$	0.91	
15	$0.026 \pm 6.23 \times 10^{-4}$	0.88	_	_	
20	_	_	$0.021 \pm 3.03 \times 10^{-4}$	0.92	
27	$0.027 \pm 5.41 \times 10^{-4}$	0.93	$0.021 \pm 4.41 \times 10^{-4}$	0.84	
50	_	_	$0.022 \pm 3.85 \times 10^{-4}$	0.90	
54	$0.023{\pm}5.11{\times}10^{-4}$	0.87	_	_	
9 0 1					

^a Standard error.

treated orange juice. The respective activation energy values were determined as 78.6 kJ/mol ($R^2=0.98$) and 62.4 kJ/mol ($R^2=0.97$). Higher activation energy was found in the case of high pressure treated juice, suggesting greater temperature dependence of the browning rate compared to that of thermally pasteurised one.

The % colour change of both high pressure and thermally treated orange juice at different temperature and time storage conditions was found to correlate linearly with the corresponding % ascorbic acid loss. The same linear dependence of % colour change on % ascorbic acid loss was found for both treatments, as illustrated in Fig. 6 (R^2 =0.85).

3.5. Rheological behaviour of orange juice

The orange juice studied was found to have a pseudoplastic rheological behaviour. The apparent viscosity, μ_{φ} (Pa · s), was described by a power law relationship (Eq. (5)), in agreement with previous findings (Polydera et al., 2003; Telis-Romero, Telis, & Yamashita, 1999):

$$\mu_{\omega} = K \cdot \gamma^{n-1} \tag{5}$$

where μ_{φ} : shear stress (Pa), γ : shear rate (s⁻¹), K: consistency index (Pa · sⁿ) and n: flow behaviour index.

The consistency index, K, and the flow behaviour index, n, were determined through non-linear regression (SYSTAT[®])



Fig. 7. Effect of storage time on apparent viscosity of high pressure treated orange juice during storage at 10 $^{\circ}$ C.

Table 6

Consistency index, K (Pa \cdot sⁿ), of high pressurised and thermally pasteurised orange juice as a function of storage temperature after a period of storage (15 or 27 days)

Storage	High pressurised juice				Thermally pasteurised juice	
temperature(°C) 15 days		27 days			27 days	
	K (Pa · s ⁿ)	R^2	$K (\operatorname{Pa} \cdot \operatorname{s}^n)$	R^2	$K (\operatorname{Pa} \cdot \operatorname{s}^n)$	R^2
0	$0.058 \pm 3.72 \times 10^{-3a}$	0.87	$0.035 \pm 1.04 \times 10^{-3}$	0.92	$0.022 \pm 3.36 \times 10^{-4}$	0.92
5	$0.042 \pm 1.87 \times 10^{-3}$	0.82	$0.029 \pm 4.57 \times 10^{-4}$	0.96	$0.021 \pm 4.29 \times 10^{-4}$	0.86
10	$0.026 \pm 6.23 \times 10^{-4}$	0.88	$0.027 \pm 5.41 \times 10^{-4}$	0.93	$0.021 \pm 4.41 \times 10^{-4}$	0.84
15	$0.024 \pm 5.36 \times 10^{-4}$	0.90	$0.031 \pm 1.17 \times 10^{-3}$	0.88	$0.022\pm5.04 imes10^{-4}$	0.81
30	$0.027 \pm 7.12 \times 10^{-4}$	0.85	-	_	_	_

^a Standard error.

8.0 Statistics, 1998, SPSS, Chicago, IL, USA) using the experimental data and Eq. (5) for both high pressurised and thermally treated orange juice at different days of storage at 0, 5, 10, 15 and 30 °C. An average flow behaviour index of about 0.587 and 0.655 was found for high pressurised and thermally treated orange juice, respectively, when all data were used simultaneously for each process. The above values indicated greater deviation of high pressurised juice from Newtonian behaviour. The consistency index values at different storage conditions were determined from Eq. (5) for the average values of the flow behaviour index for both juices and presented in Table 5 for storage at 10 °C.

A small decrease of the consistency index, which also means a decrease of the corresponding apparent viscosity values, was observed during storage of juices at all different conditions. The decrease mentioned above was more pronounced in the case of high pressurised orange juice, while the consistency index of thermally treated juice did not significantly change with storage time. The apparent viscosity curves for storage at 10 °C are illustrated indicatively in Fig. 7 for high pressure treated juice. A limited cloud loss during storage of orange juice—due to the small percentage of the remaining PME activity after processing-may be related to the decrease of the consistency index as a function of storage time. Slightly higher apparent viscosity values were determined for high pressurised orange juice compared to thermally treated one immediately after processing and at each storage day for all storage temperatures studied (Tables 5 and 6). Increase of storage temperature led to a small decrease of consistency index of high pressurised juice, as shown in Table 6. In the case of thermal pasteurisation of orange juice, storage temperature did not seem to affect the consistency index (Table 6).

4. Conclusions

A high pressure treatment of 600 MPa at 40 °C for 4 min led to a better retention of ascorbic acid during post processing storage of fresh orange juice at 0-30 °C compared to conventional thermal pasteurisation (80 °C, 60 s). An extension of shelf life was therefore achieved for high pressure treated orange juice. Immediately after processing, high pressurised orange juice retained better the flavour of untreated fresh juice, while its sensory characteristics were also judged superior during storage compared to thermally pasteurised juice. Colour change was found to correlate linearly with ascorbic acid loss. Slightly higher viscosity values of orange juice were also found when HHP was applied. Due to the above described benefits of extension of shelf life, superior organoleptic quality and better nutrient retention, high pressure technology emerges as an advantageous alternative process for high valued products like orange juice.

References

- Basak, S., Ramaswamy, H. S., & Simpson, B. K. (2001). High pressure inactivation of pectin methyl esterase in orange juice using combination treatments. *Journal of Food Biochemistry*, 25(6), 509–526.
- Boff, J. M., Truong, T. T., Min, D. B., & Shellhammer, T. H. (2003). Effect of thermal processing and carbon dioxide-assisted high pressure processing on pectinmethylesterase and chemical changes in orange juice. *Journal of Food Science*, 68(4), 1179–1184.
- Cameron, R. G., Baker, R. A., & Grohmann, K. (1997). Citrus tissue extracts affect juice cloud stability. *Journal of Food Science*, 62(2), 242–245.
- Cameron, R. G., Baker, R. A., & Grohmann, K. (1998). Multiple forms of pectinmethylesterase from citrus peel and their effects on juice cloud stability. *Journal of Food Science*, 63, 253–256.
- Cano, M. P., Hernández, A., & De Ancos, B. (1997). High pressure and temperature effects on enzyme inactivation in strawberry and orange products. *Journal of Food Science*, 62(1), 85–88.
- Cheah, P. B., & Ledward, D. A. (1995). High-pressure effects on lipid oxidation. JAOCS, 72(9), 1059–1063.
- Cheah, P. B., & Ledward, D. A. (1997). Catalytic mechanism of lipid oxidation following high pressure treatment in pork fat and meat. *Journal of Food Science*, 62, 1135–1138.
- Donsi, G., Ferrari, G., & Di Matteo, M. (1996). High pressure stabilization of orange juice: Evaluation of the effects of process conditions. *Italian Journal of Food Sciences*, 8(2), 99–106.
- Fernández-García, A., Butz, P., Bognar, A., & Tauscher, B. (2001). Antioxidative capacity, nutrient content and sensory quality of orange juice and an orange-lemon-carrot juice product after high pressure treatment and storage in different packaging. *European Food Research* and Technology, 213(4–5), 290–296.
- Goodner, J. K., Braddock, R. J., & Parish, M. E. (1998). Inactivation of pectinesterase in orange and grapefruit juices by high pressure. *Journal* of Agricultural and Food Chemistry, 46, 1997–2000.

- Goyle, A., & Ojha, P. (1998). Effect of storage on vitamin C, microbial load and sensory attributes of orange juice. *Journal of Food Science and Technology*, 35(4), 346–348.
- ISO (1991). Sensory analysis-methodology-method of investigating sensitivity of taste. ISO Standard 3972. Geneva, Switzerland: International Organization for Standarization.
- Kennedy, J. F., Rivera, Z. S., Lloyd, L. L., Warner, F. P., & Jumel, K. (1992). L-ascorbic acid stability in aseptically processed orange juice in Tetrabrik cartons and the effect of oxygen. *Food Chemistry*, 45(5), 327–331.
- Lee, H. S., & Chen, C. S. (1998). Rates of vitamin C loss and discoloration in clear orange juice concentrate during storage at temperatures of 4–24 °C. *Journal of Agricultural and Food Chemistry*, *46*(11), 4723–4727.
- Lee, H. S., & Coates, G. A. (1999). Vitamin C in frozen, fresh squeezed, unpasteurized, polyethylene-bottled orange juice: A storage study. *Food Chemistry*, 65, 165–168.
- Linton, M., McClements, J. M. J., & Patterson, M. F. (1999). Inactivation of *Escherichia coli* O157:H7 in orange juice using a combination of high pressure and mild heat. *Journal of Food Protection*, 62(3), 277–279.
- Manso, M. C., Oliveira, F. A. R., Oliveira, J. C., & Frias, J. M. (2001). Modelling ascorbic acid thermal degradation and browning in orange juice under aerobic conditions. *International Journal of Food Science & Technology*, 36(3), 303–312.
- Nienaber, U., & Shellhammer, T. H. (2001a). High-pressure processing of orange juice: Kinetics of pectinmethylesterase inactivation. *Journal of Food Science*, 66(2), 328–331.
- Nienaber, U., & Shellhammer, T. H. (2001b). High-pressure processing of orange juice: Combination treatments and a shelf life study. *Journal* of Food Science, 66(2), 332–336.
- Oruña-Concha, M. J., Gonzalez-Castro, M. J., Lopez-Hernandez, J., & Simal-Lozano, J. (1998). Monitoring of the vitamin C content of frozen green beans and Padrón peppers by HPLC. *Journal of the Science of Food and Agriculture*, 76, 477–480.
- Parish, M. E. (1998a). High pressure inactivation of *Saccharomyces cerevisiae*, endogenous microflora and pectinmethylesterase in orange juice. *Journal of Food Safety*, 18(1), 57–65.
- Parish, M. E. (1998b). Orange juice quality after treatment by thermal pasteurization or isostatic high pressure. *Lebensmittel-Wissenschaft und Technologie-Food Science and Technology*, 31, 439–442.
- Polydera, A. C., Galanou, E., Stoforos, N. G., & Taoukis, P. S. (2004a). Inactivation kinetics of pectin methylesterase of greek Navel orange juice as a function of high hydrostatic pressure and temperature process conditions. *Journal of Food Engineering*, 62(3), 291–298.
- Polydera, A. C., Stoforos, N. G., & Taoukis, P. S. (2003). Comparative shelf life study and vitamin C loss kinetics in pasteurised and high

pressure processed reconstituted orange juice. Journal of Food Engineering, 60(1), 21-29.

- Polydera, A. C., Stoforos, N. G., & Taoukis, P. S. (2004b). Effect of high hydrostatic pressure treatment on post processing antioxidant activity of fresh Navel orange juice. *Food Chemistry* (in press).
- Roig, M. G., Bello, J. F., Rivera, Z. S., & Kennedy, J. F. (1999). Studies on the occurrence of non-enzymatic browning during storage of citrus juice. *Food Research International*, 32, 609–619.
- Sadler, G., Parish, M., Van Clief, D., & Davis, J. (1997). The effect of volatile absorption by packaging polymers on flavor, microorganisms and ascorbic acid in reconstituted orange juice. *Lebensmittel-Wissenschaft und Technologie-Food Science and Technology*, 30, 686–690.
- Snir, R., Koehler, P. E., Sims, K. A., & Wicker, L. (1996). Total and thermostable pectinesterases in citrus juices. *Journal of Food Science*, 61(2), 379–382.
- Tawfik, M. S., & Huyghebaert, A. (1998). Effect of storage temperature, time, dissolved oxygen and packaging materials on the quality of aseptically filled orange juice. *Acta Alimentaria*, 27(3), 231–244.
- Telis-Romero, J., Telis, V. R. N., & Yamashita, F. (1999). Friction factors and rheological properties of orange juice. *Journal of Food Engineering*, 40, 101–106.
- Van den Broeck, I., Ludikhuyze, L. R., Van Loey, A. M., & Hendrickx, M. E. (2000). Inactivation of orange pectinesterase by combined high pressure and temperature treatments: A kinetic study. *Journal of Agricultural and Food Chemistry*, 48(5), 1960–1970.
- Versteeg, C., Rombouts, F. M., Spaansen, C. H., & Pilnik, W. (1980). Thermostability and orange juice cloud destabilizing properties of multiple pectinesterases from orange. *Journal of Food Science*, 45, 969–971.
- Weemaes, C., Ludikhuyze, L., Van den Broeck, I., & Hendrickx, M. (1999). Kinetic study of antibrowning agents and pressure inactivation of avocado polyphenoloxidase. *Journal of Food Science*, 64(5), 823–827.
- Yeom, H. W., Streaker, C. B., Zhang, Q. H., & Min, D. B. (2000). Effects of pulsed electric fields on the quality of orange juice and comparison with heat pasteurization. *Journal of Agricultural and Food Chemistry*, 48(10), 4597–4605.
- Zook, C. D., Parish, M. E., Braddock, R. J., & Balaban, M. O. (1999). High pressure inactivation kinetics of *Saccharomyces cerevisiae* ascospores in orange and apple juices. *Journal of Food Science*, 64(3), 533–535.