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Effect of ultrasound followed by high pressure processing on prebiotic cranberry juice

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ABSTRACT

This work evaluated the effect of high pressure processing (HPP) and ultrasound (US) on the quality of prebiotic cranberry juice fortified with fructo-oligosaccharides (FOS). The juice was subjected to HPP for 5 min (450 MPa) and to ultrasonic treatment for 5 min (600 and 1200 W/L) followed by HPP for 5 min (450 MPa). Chemical analyses were carried out to identify and quantify the anthocyanins, and to quantify FOS, organic acids, instrumental color, soluble solids, pH and antioxidant capacity. Both non-thermal treatments preserved the FOS content maintaining the prebiotic property of the juice. The retention of organic acids was high (>90%) and an increase in anthocyanin content (up to 24%) was observed when ultrasound was followed by HPP. The changes in instrumental color, soluble solids content and pH were negligible. The use of HPP and ultrasound processing has been proven satisfactory to treat prebiotic cranberry juice.

1. Introduction

The increasing concern for health and well-being has increased the demand for functional foods (Cruz et al., 2010). Production of functional food can be done by the addition of bioactive compounds, such as fructo-oligosaccharides (FOS), to food products (Cruz et al., 2013). Nowadays, the most foods containing prebiotic oligosaccharides are dairy based, but there is a demand for the production of non-dairy prebiotic foods (Renuka, Kulkarni, Vijayanand, & Prapulla, 2009).

Fruit juices are a suitable base for the development of functional foods because they are rich in nutrients, vitamins, fibers, minerals and antioxidants. Also, they have nice flavor profiles and are regularly consumed by all age groups, due to its refreshing taste and its status as a healthy food (Pimentel, Madrona, & Prudencio, 2014). Cranberries contain bioactive compounds such as anthocyanins, flavonoids, and condensed tannins, making them a choice for the production of a prebiotic fruit juice. In addition, cranberries have antimicrobial activity against food pathogens, and *in vitro* anticancer effect (Seeram, Adams, Hardy, & Heber, 2004; Vu et al., 2012).

Fruit juices are easily spoiled, and preservation processes are required to prolong their shelf life. Thermal treatment is the standard technique used to preserve fruit juices, but heating leads to browning reactions, instrumental color change, sugar, and vitamin loss, with an overall loss of the quality of fruit juices (Damasceno, Fernandes, Magalhães, & Brito, 2008). Non-thermal technologies have been reported as suitable options to produce juices with good sensory attributes and high nutritional content (Misra et al., 2015; Zulueta, Barba, Esteve, & Frígola, 2013).

Preservation of foods by non-thermal technologies, such as high pressure processing (HPP) and ultrasound (US), are attractive alternatives because they tend to infer minimal effects on the food quality and taste (Cullen, Tiwari, & Valdramidis, 2012). HPP has been studied for fruit juices processing, and it is already used for many industrial applications (Barba, Esteve, & Frigola, 2013; Cao et al., 2011; Evelyn, Kim, & Silva, 2016). US has been pointed out as a promising technology for food processing, especially when combined with other thermal or non-thermal technologies (Evelyn et al., 2016; Terefe, Matthies, Simons, & Versteeg, 2009).

Although HPP and US are said to infer minimal effects on the food quality and taste, most published papers are related to microbial inactivation and few studies have addressed the effects of these technologies on product quality. Thus, the objective of this study is to evaluate the effect of HPP and ultrasound processing

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followed by HPP on the bioactive compounds and quality of prebiotic cranberry juice.

2. Material and methods

2.1. Cranberry juice and sample preparation

Cranberry juice (Squeez©, Fruit Juices Ltd, Ireland) was purchased from a local supermarket (Dublin, Ireland). FOS (ORAFTI© P95) was donated by Beneo GmbH (Mann, Germany) and contains fructo-oligosaccharides with a degree of polymerization (DP) from 3 to 7, and 5% of glucose and fructose. The prebiotic cranberry juice was prepared by adding of fructo-oligosaccharide to the juice, which allows a proper intake (14 g of FOS in a 200 mL portion). The daily intake to achieve the desired prebiotic effect ranges from 5 to 15 g/day (Meyer & Stasse-Wolthuis, 2009).

2.2. Ultrasound treatment

The prebiotic cranberry juice was subjected to ultrasound using a probe ultrasound equipment (Branson Ultrasonics, 18 kHz, 500 W; Dublin, Ireland). Ultrasound processing was carried out under two different power levels, corresponding to ultrasonic power densities of 600 and 1200 W/L. The treatments were carried out for 5 min at room temperature. The increase in temperature after ultrasound processing was 3 ± 1 °C and it can be considered insignificant. US processing was carried out in triplicate.

2.3. High pressure processing

High pressure processing (HPP) was carried out in an industrial equipment (Hiperbaric model 300). Prebiotic cranberry juice was packed in 250 mL polyethylene bottles, which were placed in polyethylene plastic bags, and vacuum sealed for the high pressure processing. A control sample, containing the same concentration of FOS dissolved in water was prepared and packed in the same way as that of the prebiotic cranberry juice.

HPP was carried at HPP Tolling Business Facility (Dublin, Ireland) under the conditions applied commercially to stabilize fruit juices: 5 min at 450 MPa. The desired pressure was reached in 4 s and the depressurization was instantaneous (<1 s). The temperature in the pressure chamber was controlled at 11.5 °C during the entire process. The studies on HPP fruit juice processing are usually done in small equipment with small volumes. In the present study, industrial equipment was used, and real HPP conditions were applied. HPP was carried out in duplicate.

2.4. Thin layer chromatography analysis

The prebiotic cranberry juice samples were diluted in water (1:2 v/v), cleaned on a C-18 SPE cartridge and filtered using a cellulose acetate membrane (0.45 μM, 13 mm in diameter).

Thin Layer Chromatography (TLC) analysis was used to qualify and quantify the FOS. Silica gel TLC plates (20.0 × 20.0 cm, 60 Å medium pore diameter; product number: 99570-25EA, SIGMA-ALDRICH) were used. Samples of 1 μL were applied on the plate at 1.0 cm from the bottom and at a separation distance of 1.0 cm from each other. The plates were pre-conditioned at room temperature and then placed in the TLC chamber. The solvent system used to separate the carbohydrate mixture was n-butanol/2-propanol/H_2O (10:5:4 v/v/v) mixture (Shiomi, Onodera, & Sakai, 1997). The TLC plate was developed three times by the solvent system. The carbohydrates were revealed by spraying the plates with a solution containing butanol (80% w/w), phosphoric acid (6.78 mL), urea (3 g) and ethanol (8 mL) in 100 mL. The plates were heated at 120 °C for 10 min in an air-circulating oven. The quantification of the oligosaccharides was attained using a TLC scanner CAMAG 4 densitometer and Planar winCATS Chromatography Manager software. The wavelength used was 450 nm. The analysis was done in triplicate.

2.5. Determination of pH, total soluble solids (°Brix) and instrumental color parameters

The pH of the juice was determined by direct measurement in a 420A potentiometer (Orion Research Inc., Beverly, USA). The potentiometer was calibrated with buffer solutions of pH 4.0, 7.0 and 10.0 before use.

Total soluble solids were estimated as °Brix with an Abbe refractometer (WYA-2W, Shanghai Precision & Scientific Instrument Co. Ltd., China). The analysis was carried out at room temperature (25 ± 1 °C).

The instrumental color of the samples was measured using a Minolta colorimeter (Color Quest XE Hunter Lab, Northants, UK), which was calibrated using a white standard ($L^* = 93.97$, $a^* = 0.88$ and $b^* = 1.21$). The instrumental color was read on CIELAB scale (L^*, a^* and b^*). The analysis was done in triplicate. The L, a, b parameters were used to calculate the chromaticity and the hue angle according to Eqs. (1) and (2), respectively:

$$\text{Chroma} = \sqrt{(a^*)^2 + (b^*)^2} \quad (1)$$

$$\text{Hue} = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (2)$$

where L_o, a_o and b_o are the instrumental color values for the juice used as a control sample.

2.6. Organic acids quantification

The samples were diluted in water (1:4 v/v), filtered with glass fiber filters (AP25, 13 mm diameter, Merck Millipore Ltd.), cleaned up in a C-18 SPE cartridge, and filtered again using an 0.45 μm cellulose acetate membrane (13 mm in diameter, Merck Millipores Ltd).

High Performance Liquid Chromatographic (HPLC) analysis was used for the organic acids analysis. An HPLC system (Agilent Technologies 1260 Infinity) equipped with a quaternary pump system and a UV-DAD detector monitored at 210 nm was used. Organic acids were analyzed using an Aminex HPX-87H column (300 × 7.8 mm, Bio-Rad) at 50 °C. The elution in isocratic mode was performed with 0.01 mol/L sulfuric acid in deionized water as mobile phase for 30 min at 0.6 mL/min. The injection volume was 20 μL. The analysis was done in triplicate using Sigma-Aldrich standards of malic, quinic and citric acid.

2.7. Anthocyanins identification and quantification

Before the analysis, the samples were filtered using a cellulose acetate membrane (0.45 μm; 13 mm in diameter; Merck Millipore Ltd.). No clean-up was done to avoid the anthocyanins removal from the sample. UPLC-qTOF-MS was used to identify the anthocyanins present in the cranberry juice. The analysis was performed on an Acquity UPLC system (Waters) coupled with a Quadrupole/TOF system (Waters). A Waters Acquity UPLC BEH column (150 × 2.1 mm, 1.8 μm) was used for the analysis, with the column temperature set at 40 °C. Mobile phases were water with 0.1% of formic acid (A) and acetonitrile with 0.1% of formic acid (B). The gradient used consisted: (0–22.2) min, 12–25% B; (22.3–33.4) min, 25.1–60% B; (33.5–34.0) min, 100% B; (34.1–35.5) min, 12% B; (35.6–37.5) min, 12% B. The flow rate was 0.300 mL/min. The

injection volume of sample was 5 μL. The analysis was in ESI$^+$ mode and acquired from 110 to 1180 Da. The source temperature was 120 °C; the desolvation temperature was 350 °C and desolvation gas flow was set at 500 L/h. Leucine enkephalin was used as lock mass. The acquisition mode was MSE. The instrument was controlled by Masslynx 4.1 software (Waters Corporation).

High Performance Liquid Chromatography was used to quantify the anthocyanins. Before the analysis, the anthocyanins were extracted on a C-18 SPE cartridge (500 mg/ 6 mL) using methanol for elution and filtered in a cellulose ester acetate membrane (0.45 μm, 13 mm in diameter).

An HPLC system (Agilent Technologies 1260 Infinity) equipped with a quaternary pump system and a UV-DAD detector monitored at 530 nm was used. The anthocyanins were separated in a Shim-pack CLC-ODS column (150 × 4.6 mm, Shimadzu) at 20 °C. The mobile phase for gradient elution consisted of 10% (v/v) aqueous formic acid (solvent A) and 10% (v/v) formic acid in methanol (solvent B). The gradient was linear from 12% of solvent B to 25% of solvent B over 32 min, then linear to 60% of solvent B at 48 min, then linear to 100% of solvent B at 50 min and held there for 1 min. It was then reduced back to 12% of solvent B at 55 min. The flow rate was 1 mL/min. The injection volume was 20 μL. Quantification was performed based on DAD data. The treatments were performed in triplicate.

An external standard curve of cyanidin-3-*O*-glucoside (Cy3Glu) was used, and the concentrations were expressed as Cy3Glu equivalents (Brito et al., 2007). The calibration curve of Cy3Glu was built in a concentration range from 0.9 to 18 μg/mL.

2.8. Antioxidant activity

The antioxidant activity was assayed according to DPPH, ABTS and FRAP methods. DPPH-RSA of samples was determined by spectrophotometric analysis as described by Paz et al. (2014). Briefly, 50 μL of sample was mixed with 950 μL of 0.12 mmol/L DPPH (methanolic solution). The decrease of the absorbance was measured at 517 nm (25 °C) until stable absorption was reached and compared against the stable radical DPPH. A UV–vis spectrophotometer (Spectrum model SP200UV; Shanghai, China) was used, and the antioxidant activity was expressed as Trolox equivalent (TE). Analyses were done in triplicate.

The FRAP assay was carried out according to Gonzalez-Centeno et al. (2012). The fresh working FRAP reagent was prepared mixing 0.01 mol/L TPTZ in 0.04 mol/L HCl, 0.02 mol/L $FeCl_3$ aqueous solution and acetate buffer (pH: 3.6) at a ratio of 1:1:10 (v/v/v). For the antioxidant activity assay, 50 μL of sample was mixed with 950 μL of the FRAP reagent. The increase in absorbance was measured at 593 nm (37 °C) until stabilization and compared against the stable FRAP reagent. A UV–vis spectrophotometer (Spectrum model SP200UV; Shanghai, China) was used, and the antioxidant activity was expressed as ascorbic acid equivalent (AAE). Analyses were done in triplicate.

The ABTS assay was performed according to the methodology proposed by Santacatalina et al. (2014). The ABTS radical cation (ABTS$^{\circ+}$) was prepared by the reaction of equivalent volumes (1:1) of 7 mmol/L ABTS and 2.45 mmol/L potassium persulfate aqueous solution. This stock solution was allowed to react for 12–16 h in the dark. Then, 8 mL of the ABTS solution was diluted with EtOH/H_2O (25:75 v/v) to a final volume of 100 mL. For the measurement of the antioxidant activity, 50 μL of the sample was mixed with 950 μL of the ABTS reagent. The decrease in absorbance was measured at 734 nm (25 °C) and compared against the stable ABTS reagent. A UV–vis spectrophotometer (Spectrum model SP200UV; Shanghai, China) was used and the antioxidant activity was expressed as Trolox equivalent (TE). Analysis was done in triplicate.

2.9. Statistical analysis

Statistical analyses were performed using the statistical software Statistica (Statsoft, Tulsa, USA) version 13.0. The results were compared by one-way ANOVA analysis and Tukey test at a 95.0% confidence level. The results were reported as mean ± standard deviation (Granato, Calado, & Jarvis, 2014).

3. Results and discussion

3.1. Effect of processing on FOS

Fig. 1 presents the TLC chromatograms of FOS after high pressure processing. The results were expressed as the relative concentration of FOS obtained by densitometry. The blank refers to FOS diluted in water and the reference refers to cranberry juice processed only by HPP.

After HPP and US + HPP slight changes were observed in the FOS profile compared to the blank. A small decrease of FOS with DP5 and DP6 was observed accompanied by a slight increase in FOS with DP3 and DP4, suggesting some FOS hydrolysis. Although

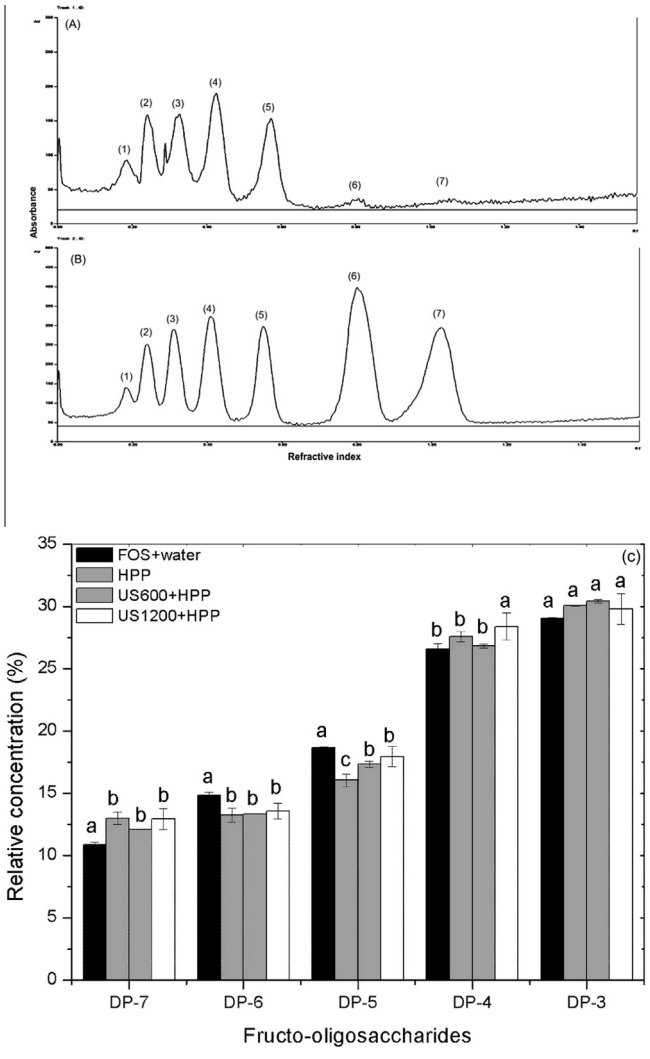

Fig. 1. TLC chromatograms of fructo-oligosaccharides subjected to high pressure processing: (A) FOS dissolved in water; (B) FOS dissolved in cranberry juice. Numbers refer to: DP7 (1), DP6 (2), DP5 – 1-fructofuranosylnystose (3), DP4 – nystose (4), DP3 – kestose (5), sucrose (6), glucose + fructose (7) and (C) relative concentration (%) of fructo-oligosaccharides in water and in cranberry juice after high pressure processing (HPP) and ultrasound processing followed by HPP.

statistical differences were observed in the FOS profile, these changes do not impart significant degradation of FOS, even when US processing was applied before HPP.

Depolymerization of oligosaccharides and polysaccharides after ultrasound processing has been reported by some studies (Hosseini et al., 2013; Zhang et al., 2016). The depolymerization caused by ultrasound application is usually attributed to the cavitation promoted by ultrasound and may involve two possible mechanisms. One mechanism is the degradation of the polymer from collapsed bubble cavitation, while the second mechanism is the chemical degradation as a result of the chemical reaction between the polymer and high-energy molecules such as hydroxyl radicals produced during cavitation.

Previous studies have shown that thermal technologies are not feasible for the production of prebiotic foods because FOS degrades at high temperatures (Forgo, Kiss, Korózs, & Rapi, 2013) and are also highly susceptible to hydrolysis during pasteurization (Klewicki, 2007; Matusek, Merész, Le, & Örsi, 2009). Non-thermal technologies, on the other hand, may be used. Almeida et al. (2016) showed that atmospheric cold plasma and ozone processing of prebiotic orange juice have partially degraded gluco-oligosaccharides, but the juice still kept a sufficient amount of oligosaccharides to be classified as a prebiotic food.

The results presented herein showed that HPP or the combination of non-thermal technologies (US + HPP) are viable processes for the treatment of prebiotic juices. A conservative prebiotic content was obtained despite the slight changes inferred during the processing.

3.2. Effect of non-thermal treatments on pH, soluble solids content, and instrumental color

The pH, instrumental color and soluble solids content of the samples treated by high pressure processing and ultrasound are shown in Table 1. The treatments did not induce any significant change in the pH and soluble solids content of prebiotic cranberry juice.

Similar results regarding pH and soluble solids content were observed in tomato, orange and apple juices treated with ultrasound (Adekunte, Tiwari, Cullen, Scannell, & O'Donnell, 2010; Tiwari, Muthukumarappan, O'Donnell, & Cullen, 2008; Zhang, Zhang, Chen, Zhang, & Hua, 2012). The pH values and soluble solids content of asparagus juice were not significantly affected by HPP when compared to thermal processing (121 °C, 3 min) and the raw juice (Chen et al., 2015).

The L parameter, which indicates the luminosity of the product, was close for all treatments: HPP (48.68 ± 0.07), US600 + HPP (49.86 ± 0.03) and US1200 + HPP (47.92 ± 0.09) indicating good preservation of luminosity. The same trend was observed for chroma and hue, which indicates the vivacity of color and the color characteristic. Thus, cranberry juice kept its vivacity and characteristic color after processing. Although significant statistical differences were obtained for L, C and h (Table 1), these differences are caused by the small standard deviation of the measurements. However, the human eye does not perceive those of small color variations among samples. Barba et al. (2013) also concluded that color changes were not visually noticeable after HPP of blueberry juice (200, 400 and 600 MPa; 5, 9 and 15 min).

3.3. Organic acids quantification

Non-thermal processing has not shown significant differences in the concentration of the organic acids. The concentration of malic acid after HPP (4.45 ± 0.04 g/L) was close to the concentration of malic acid in the prebiotic cranberry juice after US600 + HPP (4.11 ± 0.02 g/L) and after US1200 + HPP (4.59 ± 0.05 g/L). The concentrations of citric acid and quinic acid also did not show large variations after HPP (1.09 ± 0.01 and 0.96 ± 0.01 g/L), US600 + HPP (0.98 ± 0.02 and 0.86 ± 0.01 g/L) and US1200 + HPP (1.21 ± 0.01 and 1.03 ± 0.03 g/L) treatments. The results obtained for the organic acids are in agreement with the changes observed in the pH values (Table 1).

The lack of trend presented by malic, citric and quinic acid in processed cranberry juice, especially due to ultrasound application, differed from the trends reported for ascorbic acid. The concentration of ascorbic acid in apple, lime and grapefruit juices have increased after ultrasound processing (30, 60 and 90 min) compared with control samples (Aadil, Zeng, Han, & Sun, 2013; Abid et al., 2013; Bhat, Kamaruddin, Min-Tze, & Karim, 2011).

The results, however, are positive, since pasteurization and pulsed electric field processing have led to significant degradation of the organic acids in fruit juices (Igual, García-Martínez, Camacho, & Martínez-Navarrete, 2010).

3.4. Anthocyanins identification and quantification

Seven anthocyanins were identified by UPLC-qTOF-MS in the cranberry juice: cyanidin-3-*O*-galactoside (Cy3Gal), cyanidin-3-*O*-glucoside (Cy3Glu), cyaniding-3-*O*-arabinoside (Cy3Ara), peonidin-*O*-3-galactoside (Peo3Gal), peonidin-3-*O*-glucoside (Peo3Glu), malvidin-3-*O*-galactoside (Mal3Gal) and peonidin-3-*O*-arabinoside (Peo3Gal). Table 2 shows the $[M+H]^+$ of the 7 anthocyanins. ESI^+ signals attributable to anthocyanins were observed in the methanolic extract of the juice. The $[M+H]^+$ ion at m/z 449.1 and the fragment m/z 287.2 were detected in both cyanidins (Cy3Glu and Cy3Gal). The $[M+H]^+$ ion at m/z 463 and the fragment m/z 301.1 were detected in both peonidins (Peo3Glu and Peo3Gal). The $[M+H]^+$ ions at m/z 419.1, 493.1 and 433.1 were assigned to cyanidin-3-*O*-arabinoside, malvidin-3-*O*-galactoside and peonidin-3-*O*-arabinoside, respectively (Fig. 2). The chromatogram of the prebiotic cranberry juice is presented in Fig. 3, showing the peaks for the 7 anthocyanins. The results obtained herein are in agreement to those obtained by Hummer, Durst, Zee, Atnip, and Giust (2013), where the cranberry profile included peaks of cyanidins and peonidins glycosylated with galactose, glucose, and arabinose.

The combination of ultrasound (1200 W/L) followed by HPP has increased the amounts of Cy3Gal, Cy3Ara, Peo3Gal, Peo3Glu and Mal3Gal in the prebiotic juice (Table 2). A decrease of Peo3Ara was observed when ultrasound was combined with HPP.

Anthocyanins are the main compounds responsible for the color of cranberry juice. The increase in anthocyanin content observed

Table 1
Values of pH, soluble solids content and color parameters of prebiotic cranberry juice subjected to high pressure processing and ultrasound treatments followed by HPP.

Samples	pH	Brix	Color parameters		
			L^*	C	h^o
HPP	2.65 ± 0.01[a]	17.7 ± 0.01[a]	48.68 ± 0.07[b]	33.42 ± 0.10[c]	25.63 ± 0.06[b]
US600 + HPP	2.65 ± 0.01[a]	17.6 ± 0.01[a]	49.86 ± 0.03[a]	33.70 ± 0.08[b]	25.37 ± 0.03[c]
US1200 + HPP	2.61 ± 0.02[b]	17.4 ± 0.02[b]	47.92 ± 0.09[b]	34.05 ± 0.04[a]	25.80 ± 0.05[a]

Values with different letters in the same column (a–c) are significantly different ($p < 0.05$).

Table 2
Concentration (μg/mL) of anthocyanins identified in prebiotic cranberry juice after high pressure processing and ultrasound treatments.

Peak	Acys	Rt (min)	$[M+H]^+$	HPP	US600 + HPP	US1200 + HPP
1	Cy3Gal	7.12	449.1	2.00 ± 0.01[a]	1.81 ± 0.06[b]	2.12 ± 0.02[a]
2	Cy3Glu	8.53	449.1	0.21 ± 0.00[a]	0.21 ± 0.01[a]	0.21 ± 0.00[a]
3	Cy3Ara	9.96	419.1	1.51 ± 0.03[b]	1.42 ± 0.03[b]	1.72 ± 0.03[a]
4	Peo3Gal	12.33	463.1	2.56 ± 0.03[b]	2.20 ± 0.09[c]	3.15 ± 0.08[a]
5	Peo3Glu	14.56	463.1	0.32 ± 0.00[b]	0.32 ± 0.00[b]	0.36 ± 0.01[a]
6	Mal3Gal	16.51	493.1	1.12 ± 0.01[b]	0.99 ± 0.01[c]	1.33 ± 0.01[a]
7	Peo3Ara	17.69	433.1	0.69 ± 0.01[a]	0.59 ± 0.00[b]	0.57 ± 0.00[b]

Average ± standard deviation of three replicates. Values with different letters in the same line (a–c) are significantly different ($p < 0.05$).

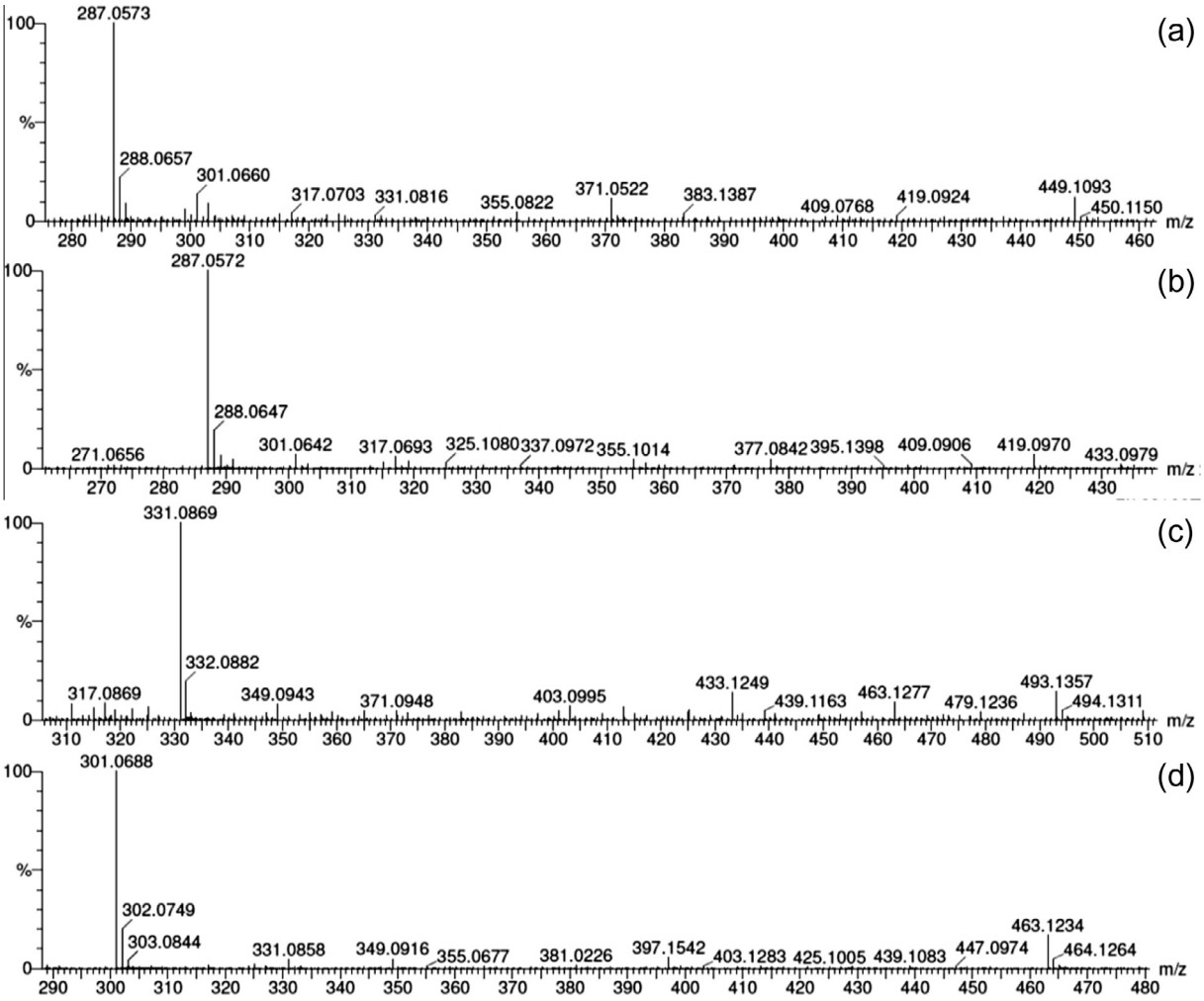

Fig. 2. Mass spectrum of the main anthocyanins identified in prebiotic cranberry juice: (a) Cy3Gal, (b) Cy3Ara, (c) Mal3Gal, (d) Peo3Glu.

when ultrasound (1200 W/L) was combined with HPP corroborates with results from instrumental color analysis, which showed an increase in chroma and decrease in luminosity, caused by the darker color of these anthocyanins (Tables 1 and 2).

The increase in pelargonidin-3-*O*-glucoside content has been reported for strawberry juice and has been attributed to the extraction of bounded anthocyanins from the suspended pulp at lower amplitude levels of ultrasound application (Tiwari, O'Donnell, Patras, & Cullen, 2008). As for Peo3Glu, the increase of Cy3Gal, Cy3Ara, Peo3Gal and Mal3Gal may also be attributed to extraction of bounded anthocyanins from the suspended pulp, since they share the same kind of structure. At lower ultrasonic power density (600 W/L), the degradation of the anthocyanins surpasses the number of bounded-anthocyanins that are being extracted and a decrease in anthocyanin content was observed (Tiwari, O'Donnell, & Cullen, 2009). A similar trend was observed in red wine, where an increase in the phenolic compounds content was found (Masuzawa, Ohdaira, & Ide, 2000).

Rodríguez-Pérez et al. (2015) studied the effect of gamma-irradiation on cranberry syrup. A significant increase in the procyanidins was observed after the processing. Chen and Martynenko (2016) reported the effect of Hydrothermodynamic (HTD) processing on anthocyanins stability. An increase of 7–13% in the anthocyanin content was reported after HTD at low temperatures (70 and 75 °C). However, anthocyanin content was negatively affected after HDT at temperatures above 80 °C.

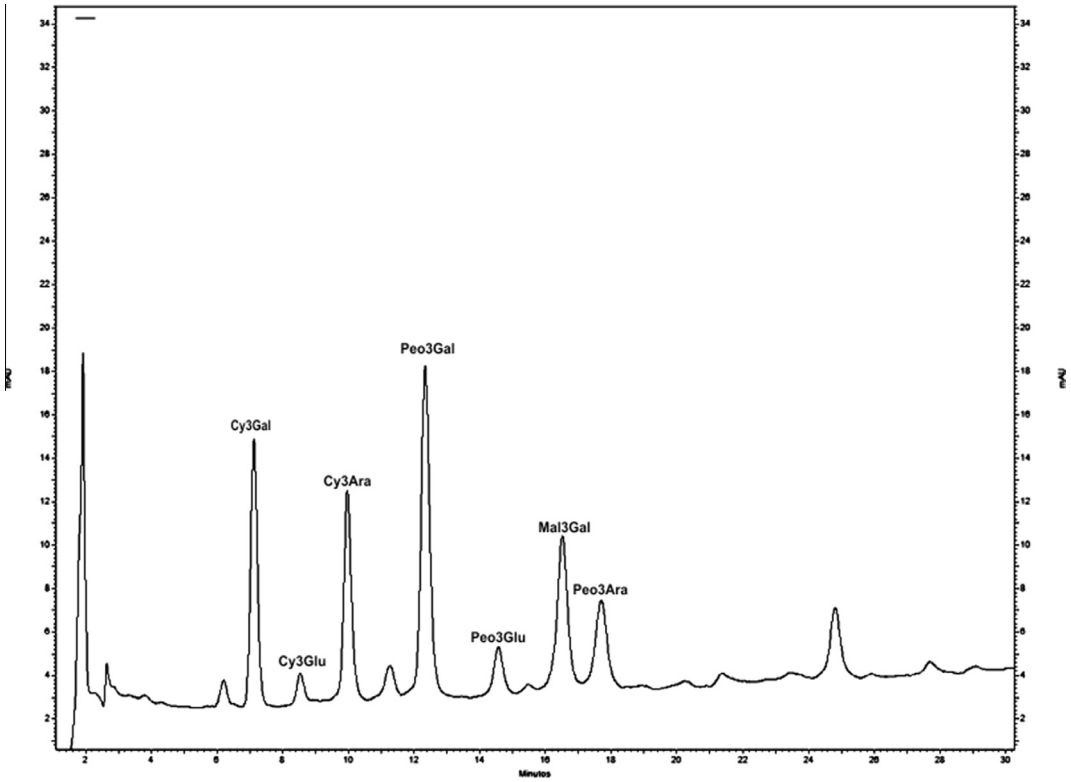

Fig. 3. HPLC chromatogram of the anthocyanins present in the prebiotic cranberry juice subjected to high pressure processing.

Anthocyanins are relatively stable and are not prone to intense degradation by ultrasound application. Studies have shown that blackberry and strawberry juice presented retention levels of anthocyanins of more than 98% (Tiwari et al., 2009).

Some studies reported the effect of thermal pasteurization and HPP processing at cold and mild temperatures on the chemical composition, microbial and enzyme activity in strawberry purée. It was observed that the concentration of anthocyanins (cyanidin-3-*O*-glucoside, pelargonidin-3-*O*-glucoside and pelargonidin-3-*O*-rutinoside) present in the strawberry purée decreased after high pressure processing (0 and 50 °C) and thermal pasteurization treatments compared to control samples (Marszałek, Mitek, & Skąpska, 2015). The same trend was observed on other fruit juices confirming the negative effect of thermal processes over the anthocyanin content (Cao et al., 2011; Marszałek, Mitek, & Skąpska, 2011).

3.5. Antioxidant capacity

Total antioxidant capacity of prebiotic cranberry juice was evaluated using DPPH, FRAP and ABTS free radical methods. The results are presented in Table 3. No statistical difference was observed among the juices subjected to HPP and to US (600 W/L) + HPP and under these conditions the juice has not lost its antioxidant potential. A decrease, however, was observed when an increase in the ultrasonic power density was applied before HPP. This decrease is mainly related to the formation of a higher amount of hydroxyl radicals during ultrasound application and consequent reaction between these radicals and the anthocyanins (Fernandes, Oliveira, Gomes, & Rodrigues, 2016). Although the antioxidant capacity decreased with the ultrasound power density increase, the antioxidant capacity was still high when compared to other fruit juices after the processing. The antioxidant capacity obtained in this study are in agreement to the reported by Granato, Karnopp, and van Ruth (2015) for the cranberry juice.

Table 3
Antioxidant activity in prebiotic cranberry juice after HPP and US + HPP treatment.

Treatment	FRAP (mg AAE/mL)	DPPH (mg TE/mL)	ABTS (mg TE/mL)
HPP	0.211 ± 0.008^{a}	$0.685 \pm 0.034^{a,b}$	0.471 ± 0.066^{a}
US600 + HPP	0.210 ± 0.006^{a}	0.703 ± 0.018^{a}	0.479 ± 0.008^{a}
US1200 + HPP	0.123 ± 0.007^{b}	0.611 ± 0.006^{b}	0.448 ± 0.062^{a}

Average ± standard deviation of three replicates. Values with different letters in the same column (a–b) are significantly different ($p < 0.05$). AAE means ascorbic acid equivalent; TE means trolox equivalent.

Caminiti et al. (2011) showed the impact of selected combinations of non-thermal processing technologies (ultraviolet light and high intensity light pulses in combination with pulsed electric fields and manothermosonication) on the quality of a blend of apple and cranberry juice. According to the authors, an adverse effect on the product quality was only observed under manothermosonication. The antioxidant capacity is a difficult parameter to be compared due to variations in the sample origin and methodology protocol. Higher antioxidant activities are usually reported for non-commercial cranberry juice, but the antioxidant capacity of the commercial cranberry juice is still high compared to other commercial fruit juices (Oszmiánski, Wojdylo, Lachiwicz, Gorzelany, & Matlok, 2016).

In functional food processing, the main concern is the maintenance of the product functionality. After the processing, the prebiotic content was not significantly affect, preserving the prebiotic claim. The combination of US + HPP on prebiotic cranberry juice showed adverse effect only in the antioxidant activity when the higher US intensity was applied, although an increase of some anthocyanins was observed.

The combination of nonthermal technologies with mild heat, antimicrobial agents and low pH to enhance the microbial inactivation in foods, has been widely described as a suitable strategy for food processing. The combination of nonthermal technologies

such as pulsed electric field (PEF) and UV-light was recently described as efficient in food processing regarding the microbial inactivation. However, the same strategy might contribute for other purposes such as processing and quality improvement (Fonteles et al., 2012; Ojha, Kerry, Alvarez, Walsh, & Tiwari, 2016)

The combination of one or more processing can be done simultaneously or sequentially, where the order of the steps leads to different results. Ultrasound processing of fruit juices improves the fruit juice quality (Costa et al., 2013). However, this technology is usually not enough to assure microbiological stability. Thus, the effect of ultrasound when used with other processing technologies (high pressure, heat and others) has been extensively studied (Sgimic & Rajkovic, 2014).

The use of ultrasound combined with other food processing can allow a better result besides energy save (Demirdöven & Baysal, 2008; Fernandes, Linhares, & Rodrigues, 2008; Garcia-Noguera, Weller, Oliveira, Rodrigues, & Fernandes, 2010). HPP might be considered an expensive non-thermal technology due to the high equipment cost. However, with the versatility of HPP equipment's, the processing can be done on demand as done with gamma irradiation. This strategy allows the small industry to the benefit of hurdle technology with lower investments. The use of US followed by HPP can be done applying the US processing in site and the HPP in a company specialized in this processing, such as HPP Tolling. This strategy is possible because HPP is the final step and it is done with the packaged product.

The consumer's acceptance (Fonseca et al., 2016; Gaze et al., 2015) and *in vitro* test are subject to further research (Lollo et al., 2015; Lollo et al., 2015).

4. Conclusion

HPP and US + HPP treatments showed promising results for prebiotic cranberry juice. Good preservation of FOS was obtained after the processing. The instrumental color analysis, pH, soluble solids content, organic acids, bioactive compounds and antioxidant capacity did not show any critical changes after the treatments. The results showed that US + HPP and high pressure processing are suitable non-thermal processing technologies to preserve the quality and functionality of prebiotic cranberry juice containing FOS.

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References

Aadil, R. M., Zeng, X., Han, Z., & Sun, D. (2013). Effects of ultrasound treatments on quality of grapefruit juice. *Food Chemistry, 141*, 3201–3206.

Abid, M., Jabbar, S., Wu, T., Hashim, M. M., Hu, B., Lei, S., ... Zeng, X. (2013). Effect of ultrasound on different quality parameters of apple juice. *Ultrasonics Sonochemistry, 20*, 1182–1187.

Adekunte, A. O., Tiwari, B. K., Cullen, P. J., Scannell, A. G. M., & O'Donnell, C. P. (2010). Effect of sonication on colour, ascorbic acid and yeast inactivation in tomato juice. *Food Chemistry, 122*, 500–507.

Almeida, F. D. L., Cavalcante, R. S., Cullen, P. J., Frias, J. M., Bourke, P., Fernandes, F. A. N., & Rodrigues, S. (2016). Effects of atmospheric cold plasma and ozone on prebiotic orange juice. *Innovative Food Science and Emerging Technologies, 32*, 127–135.

Barba, F. J., Esteve, M. J., & Frigola, A. (2013). Physicochemical and nutritional characteristics of blueberry juice after high pressure processing. *Food Research International, 50*, 545–549.

Bhat, R., Kamaruddin, N. S. B. C., Min-Tze, L., & Karim, A. A. (2011). Sonication improves kasturi lime (*Citrus microcarpa*) juice quality. *Ultrasonics Sonochemistry, 18*, 1295–1300.

Brito, E. S., Araújo, M. C. P., Alves, R. E., Carkeet, C., Clevidence, B. A., & Novotny, J. A. (2007). Anthocyanins present in selected tropical fruits: acerola, jambolão, jussara, and guajiru. *Journal of Agricultural and Food Chemistry, 55*, 9389–9394.

Caminiti, I. M., Noci, F., Muñoz, A., Whyte, P., Morgan, D. J., Cronin, D. A., & Lyng, J. G. (2011). Impact of selected combinations of non-thermal processing technologies on the quality of an apple and cranberry juice blend. *Food Chemistry, 124*, 1387–1392.

Cao, X., Zhang, Y., Zhang, F., Wang, Y., Yi, J., & Liao, X. (2011). Effects of high hydrostatic pressure on enzymes, phenolic compounds, anthocyanins, polymeric color and color of strawberry pulps. *Journal of the Science and Food Agriculture, 91*, 877–885.

Chen, Y., & Martynenko, A. (2016). Effect of hydrothermodynamic (HTD) processing on physical and chemical qualities of American cranberry puree using response surface methodology (RSM). *LWT – Food Science and Technology, 70*, 322–332.

Chen, X., Qin, W., Ma, L., Xu, F., Jin, P., & Zheng, Y. (2015). Effect of high pressure processing and thermal treatment on physicochemical parameters, antioxidant activity and volatile compounds of green asparagus juice. *LWT – Food Science and Technology, 62*, 927–933.

Costa, M. G. M., Fonteles, T. V., de Jesus, A. L. T., Almeida, F. D. L., de Miranda, M. R. A., Fernandes, F. A. N., & Rodrigues, S. (2013). High-intensity ultrasound processing of pineapple juice. *Food and Bioprocess Technology*, 997–1006.

Cruz, A. G., Cadena, R. S., Walter, E. H. M., Mortazavian, A. M., Granato, D., Faria, J. A. F., & Bolini, H. M. A. (2010). Sensory analysis: relevance for prebiotic, probiotic, and synbiotic product development. *Comprehensive Reviews in Food Science and Food Safety, 9*, 358–373.

Cruz, A. G., Cavalcanti, R. N., Guerreiro, L. M. R., Sant'Ana, A. S., Nogueira, L. C., Oliveira, C. A. F., ... Bolini, H. M. A. (2013). Developing a prebiotic yogurt: Rheological, physico-chemical and microbiological aspects and adequacy of survival analysis methodology. *Journal of Food Engineering, 114*, 323–330.

Cullen, P. J., Tiwari, B. K., & Valdramidis, V. P. (2012). Status and trends of novel thermal and non-thermal technologies for fluid foods. In V. P. Valdramidis (Ed.). *Novel Thermal and Non-Thermal Technologies for Fluid Foods* (pp. 1–6). San Diego: Academic Press.

Damasceno, L. F., Fernandes, F. A. N., Magalhães, M. M. A., & Brito, E. S. (2008). Non-enzymatic browning in clarified cashew apple juice during thermal treatment: Kinetics and process control. *Food Chemistry, 106*(1), 172–179.

Demirdöven, A., & Baysal, T. (2008). The use of ultrasound and combined technologies in food preservation. *Food Reviews International, 25*(1), 1–11.

Evelyn Kim, H. J., & Silva, F. M. V. (2016). Modeling the inactivation of *Neosartorya fischeri* ascospores in apple juice by high pressure, power ultrasound and thermal processing. *Food Control, 59*, 530–537.

Fernandes, F. A. N., Linhares, F. E., & Rodrigues, S. (2008). Ultrasound as pre-treatment for drying of pineapple. *Ultrasonics Sonochemistry, 15*(6), 1049–1054.

Fernandes, F. A. N., Oliveira, V. S., Gomes, W. F., & Rodrigues, S. (2016). Degradation kinetics of vitamin E during ultrasound application and the adjustment in avocado purée by tocopherol acetate addition. *LWT – Food Science and Technology, 69*, 342–347.

Fonseca, F. G. A., Esmerino, E. A., Filho, E. R. T., Ferraz, J. P., da Cruz, A. G., & Bolini, H. M. A. (2016). Novel and successful free comments method for sensory characterization of chocolate ice cream: A comparative study between pivot profile and comment analysis. *Journal of Dairy Science, 99*, 3408–3420.

Fonteles, T. V., Costa, M. G. M., de Jesus, A. L. T., de Miranda, M. R. A., Fernandes, F. A. N., & Rodrigues, S. (2012). Power ultrasound processing of cantaloupe melon juice: Effects on quality parameters. *Food Research International, 48*(1), 41–48.

Forgo, P., Kiss, A., Korózs, M., & Rapi, S. (2013). Thermal degradation and consequent fragmentation of widely applied oligosaccharides. *Microchemical Journal, 107*, 37–46.

Garcia-Noguera, J., Weller, C. L., Oliveira, F. I. P., Rodrigues, S., & Fernandes, F. A. N. (2010). Dual-stage sugar substitution in strawberries with a Stevia-based sweetener. *Innovative Food Science & Emerging Technologies, 11*(1), 225–230.

Gaze, L. V., Oliveira, B. R., Ferrao, L. L., Granato, D., Cavalcanti, R. N., Conte Júnior, C. A., ... Freitas, M. Q. (2015). Preference mapping of dulce de leche commercialized in Brazilian markets. *Journal of Dairy Science, 98*, 1443–1454.

Gonzalez-Centeno, M. R., Jourdes, M., Femenia, A., Simal, S., Rossello, C., & Teissedre, P. L. (2012). Proanthocyanidin composition and antioxidant potential of the stem winemaking by-products from ten different grape varieties (*Vitis vinifera* L.). *Journal of Agricultural and Food Chemistry, 60*(48), 11850–11858.

Granato, D., Calado, V. M. A., & Jarvis, B. (2014). Observations on the use of statistical methods in food science and technology. *Food Research International, 55*, 137–149.

Granato, D., Karnopp, A. R., & van Ruth, S. M. (2015). Characterization and comparison of phenolic composition, antioxidant capacity and instrumental taste profile of juices from different botanical origins. *Journal of the Science of Food and Agriculture, 95*(10), 1997–2006.

Hosseini, S. M. H., Emam-Djomeh, Z., Razavi, S. H., Moosavi-Movahedi, A. A., Saboury, A. A., Atri, M. S., & Meeren, P. V. (2013). β-Lactoglobulinesodium alginate interaction as affected by polysaccharide depolymerization using high intensity ultrasound. *Food Hydrocolloids, 32*, 235–244.

Hummer, K., Durst, R., Zee, F., Atnip, A., & Giust, M. M. (2013). Phytochemicals in fruits of Hawaiian wild cranberry relatives. *Journal of the Science of Food and Agriculture, 94*, 1530–1536.

Igual, M., García-Martínez, E., Camacho, M. M., & Martínez-Navarrete, N. (2010). Effect of thermal treatment and storage on the stability of organic acids and the functional value of grapefruit juice. *Food Chemistry, 118*, 291–299.

Klewicki, Robert (2007). The stability of gal-polyols and oligosaccharides during pasteurization at a low pH. *LWT – Food Science and Technology, 40*(7), 1259–1265.

Lollo, P. C. B., Morato, P. N., de Moura, C. S., de Oliveira, M. M., Cruz, A. G., Faria, J., ... Cristianini, M. (2015). Ultra-high temperature plus dynamic high pressure processing: An effective combination for potential probiotic fermented milk processing which attenuate exercise-induced immune suppression in Wistar rats. *Journal of Functional Foods, 14*, 541–548.

Lollo, P. C. B., Morato, P. N., Moura, C. S., Almada, C. N., Felicio, T. L., Esmerino, E. A., ... Cruz, A. G. (2015). Hypertension parameters are attenuated by the continuous consumption of probiotic Minas cheese. *Food Research International, 76*, 611–617.

Marszałek, K., Mitek, M., & Skąpska, S. (2011). Application of high hydrostatic pressure to stabilize juices and nectars. *Food and Science Technology, 1*(74), 113–123.

Marszałek, K., Mitek, M., & Skąpska, S. (2015). The effect of thermal pasteurization and high pressure processing at cold and mild temperatures on the chemical composition, microbial and enzyme activity in strawberry purée. *Innovative Food Science and Emerging Technologies, 27*, 48–56.

Masuzawa, N., Ohdaira, E., & Ide, M. (2000). Effects of ultrasonic irradiation on phenolic compounds in wine. *Japanese Journal of Applied Physics, 39*, 2978–2979.

Matusek, A., Merész, P., Le, T. K. D., & Örsi, F. (2009). Effect of temperature and pH on the degradation of fructo-oligosaccharides. *European Food Research and Technology, 228*, 355–365.

Meyer, D., & Stasse-Wolthuis, M. (2009). The bifidogenic effect of inulin and oligofructose and its consequences for gut health. *European Journal of Clinical Nutrition, 63*(11), 1277–1289.

Misra, N. N., Kaur, S., Tiwari, B. K., Kaur, A., Singh, N., & Cullen, P. J. (2015). Atmospheric pressure cold plasma (ACP) treatment of wheat flour. *Food Hydrocolloids, 44*, 115–121.

Ojha, K. S., Kerry, J. P., Alvarez, C., Walsh, D., & Tiwari, B. K. (2016). Effect of high intensity ultrasound on the fermentation profile of *Lactobacillus sakei* in a meat model system. *Ultrasonics Sonochemistry, 31*, 539–545.

Oszmiánski, J., Wojdylo, A., Lachiwicz, S., Gorzelany, J., & Matlok, N. (2016). Comparison of bioactive potential of cranberry fruit and fruit-based products versus leaves. *Journal of Functional Foods, 22*, 232–242.

Paz, M., Gúllon, P., Barroso, M. F., Carvalho, A. P., Domingues, V. F., Gomes, A. M., & Delerue-Matos, C. (2014). Brazilian fruit pulps as functional foods and additives: Evaluation of bioactive compounds. *Food Chemistry, 172*, 462–468.

Pimentel, T. C., Madrona, G. S., & Prudencio, S. H. (2014). Probiotic clarified apple juice with oligofructose or sucralose as sugar substitutes: sensory profile and acceptability. *Food Science and Technology, 62*, 1–9.

Renuka, B., Kulkarni, S. G., Vijayanand, P., & Prapulla, S. G. (2009). Fructooligosaccharide fortification of selected fruit juice beverages: effect on the quality characteristics. *LWT – Food Science and Technology, 42*(5), 1031–1033.

Rodríguez-Pérez, C., Quirantes-Piné, R., Contreras, M. M., Uberos, J., Fernández-Gutiérrez, A., & Segura-Carretero, A. (2015). Assessment of the stability of proanthocyanidins and other phenolic compounds in cranberry syrup after gamma-irradiation treatment and during storage. *Food Chemistry, 174*, 392–399.

Santacatalina, J. V., Rodríguez, O., Simal, S., Cárcel, J. A., Mulet, A., & García-Pérez, J. V. (2014). Ultrasonically enhanced low-temperature drying of apple: Influence on drying kinetics and antioxidant potential. *Journal of Food Engineering, 138*, 35–44.

Seeram, N. P., Adams, L. S., Hardy, M. L., & Heber, D. (2004). Total cranberry extract versus its phytochemical constituents: Antiproliferative and synergistic effects against human tumor cell lines. *Journal of Agricultural and Food Chemistry, 52*, 2512–2517.

Sgimic, N., & Rajkovic, A. (2014). Hurdle technology. In V. Ravishankar Rai & Jamuna A. Bai (Eds.). *Microbial Safety and Preservation Techniques* (pp. 542). Boca Raton, FL, USA: CRC Press.

Shiomi, N., Onodera, S., & Sakai, H. (1997). Fructooligosaccharide content and fructosyltransferase activity during growth of onion bulbs. *New Phytologist, 136*, 105–113.

Terefe, N. S., Matthies, K., Simons, L., & Versteeg, C. (2009). Combined high pressure-mild temperature processing for optimal retention of physical and nutritional quality of strawberries (Fragaria X ananassa). *Innovative Food Science and Emerging Technologies, 10*, 297–307.

Tiwari, B. K., Muthukumarappan, K., O'Donnell, C. P., & Cullen, P. J. (2008). Effects of sonication on the kinetics of orange juice quality parameters. *Journal of Agricultural and Food Chemistry, 56*, 2423–2428.

Tiwari, B. K., O'Donnell, C. P., & Cullen, P. J. (2009). Effect of non-thermal processing technologies on the anthocyanin content of fruit juices. *Trends in Food Science & Technology, 20*(4), 137–145.

Tiwari, B. K., O'Donnell, C. P., Patras, A., & Cullen, P. J. (2008). Anthocyanin and ascorbic acid degradation in sonicated strawberry juice. *Journal of Agricultural and Food Chemistry, 56*, 10071–10077.

Vu, K. D., Carlettini, H., Bouvet, J., Côté, J., Doyon, G., & Sylvain, J-F. (2012). Effect of different cranberry extracts and juices during cranberry juice processing on the antiproliferative activity against two colon cancer cell lines. *Food Chemistry, 132*, 959–967.

Zhang, M. F., Qin, Y. H., Ma, J. Y., Yang, L., Wu, Z. K., Wang, T. L., ... Wang, C. W. (2016). Depolymerization of microcrystalline cellulose by the combination of ultrasound and Fenton reagent. *Ultrasonics Sonochemistry, 31*, 404–408.

Zhang, Y., Zhang, Z., Chen, F., Zhang, H., & Hua, X. (2012). Effect of sonication on eliminating of phorate in apple juice. *Ultrasonics Sonochemistry, 19*, 43–48.

Zulueta, A., Barba, F. J., Esteve, M. J., & Frígola, A. (2013). Changes in quality and nutritional parameters during refrigerated storage of an orange juice–milk beverage treated by equivalent thermal and non-thermal processes for mild pasteurization. *Food and Bioprocess Technology, 6*(8), 2018–2030.