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ORIGINAL ARTICLE

Rheological properties of ultrasound treated apple, cranberry and blueberry juice and nectar

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Abstract Ultrasound is non-thermal food processing technique that has been used in food processing very extensively for the last 10 years. The objective of this study was to investigate the effect of high power ultrasound and pasteurization on rheological properties (*n* and *k*) of apple, cranberry and blueberry juice and nectar. Samples were treated according the experimental design, with high power sonicator at ultrasound frequency of 20 kHz under various conditions (treatment time, temperature of sample and amplitude). Thermosonication and sonicaton of juice and nectar samples have been performed. It was found that all samples of untreated, pasteurized and ultrasonically treated apple, cranberry and blueberry juices and nectars shows non-Newtonian dilatant fluid characteristics ($n>1$). The interaction of treatment time and temperature of sample (BC) and temperature (C) of sample of apple juice had statistically significant effect on flow behavior index (*n*) for ultrasound treated apple juice. Interaction of treatment time and temperature of sample (BC) has statistically significant effect on the flow behavior index (n) for blueberry nectar. Also, there is statistically significant effect of temperature (C) of sample on consistency coefficient (*k*) for ultrasound treated apple juice.

Keywords High power ultrasound · Fruit juice and nectar · Rheological properties

Introduction

Ultrasonication is a non-thermal method of food processing that has the advantage of preserving fruit juices without causing the common side-effects associated with conventional heat treatments (Chemat et al. 2011; Salleh-Mack and Roberts 2007; Leadley and Williams 2006; Knorr et al. 2004; Lorimer et al. 1996). Applications of ultrasound in processing of fruit juices and the effects of sonication on fruit juices have been studied (Tiwari et al. 2008a, b; Tiwari et al. 2009a, b, c, d; Rawson et al. 2011; Dubrović et al. 2011). Sonication technology can improve the process through reduced processing time, higher throughput and lower energy consumption (Piyasena et al. 2003; Patist and Bates 2008). If ultrasound were to be used in any practical application, it would most likely have to be used in conjunction with pressure treatment (manosonication), heat treatment (thermosonication) or both (manothermosonication). The effect of ultrasound has been attributed to physical effects (cavitation, mechanical effects, micro-mechanical) and/or chemical effects, due to formation of free radicals (H^+ and OH^- due to sonochemical reaction) formed by the decomposition of water inside the oscillating bubbles.

Mathematical modelling is important in reducing energy consumption, lower number of experiments and analysis of interaction between investigated parameters that cannot be considered using simple statistical analysis. Response surface methodology (RSM) may be employed to optimise critical processing parameters by estimating interactive and quadratic effects. A further benefit of using RSM is the reduction in the number of experiments needed as compared to a full experimental design (Myers and Montgomery 2002,

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Lu et al. 2008). RSM has been successfully employed to optimise food processing operations by several investigators (Tiwari et al. 2008a; Rawson et al. 2011). Process optimization of thermal processes in combination with non-thermal technologies such as high pressure, ultrasound, pulsed electric field has significant potential. In study by Tiwari et al. (2008c), freshly squeezed orange juice was subjected to sonication at amplitude levels ranging from 40 % to 100 % at a constant frequency of 20 kHz. The model predictions for critical quality parameters of Hunter colour values (L*, a* and b*), cloud value and browning index were closely correlated to the experimental results obtained. RSM was demonstrated to be an effective technique to model the effect of sonication on juice quality while minimising the number of experiments required. In study by Fonteles et al. (2012) the effects of ultrasound process on quality parameters and on enzyme activities of cantaloupe melon juice were investigated. A factorial central composite design was carried out changing processing time and ultrasound intensity. The technology showed to be suitable for cantaloupe melon stabilization as alternative to thermal and other treatments that results in quality loss.

Fruit products found in the market undergo commercial processing techniques most of which are thermal processing though some of the industries may have products processed by non thermal processing. The food processing environment poses a number of challenges to obtaining rheological measurements. Rotational, vibrational and tube viscometers are the more common types of traditional measuring techniques based upon controlled deformation of the sample (Cullen et al. 2000). In study by Wu et al. (2008) tomato juice was subjected to thermosonication (TS) (24 kHz), at amplitudes of 25, 50 and 75 μm at 60, 65 and 70 °C or heat only treatments. All samples treated by termosonication (TS) had a greater apparent viscosity than the heat treated samples. In study by Vercet et al. (2002) the effect of manothermosonication (MTS), the simultaneous application of heat and high energy ultrasound waves under moderate pressure, on tomato juice rheology and on tomato enzyme activity in tomato juice was examined. The results suggest that manothermosonication (MTS) could be a useful technology to obtain high viscosity and consistency of tomato juice. The low viscosity of the juice makes pumping and evaporative concentration easier and reduces fouling of heat exchangers.

The purpose of this investigation was to examine the influence of high power ultrasound and pasteurisation on rheological properties of apple, cranberry, blueberry juice and nectars. Optimization of ultrasound parameters and validation of model for remaining rheological properties have been calculated.

Materials and methods

Juice and nectar preparation

Based on national Regulation for production of fruit juices and complementary products, two different apple, cranberry and blueberry juices have been made. Under national regulations manufacturers of fruit juices must meet demands from legislative about what percentage of fruit components must be in beverage from specific fruit, than percentage of sugar and minimal °Bx that must meet the conditions of Regulations. According to meet the demand fruit juices and beverages were produced. 100 % apple, cranberry and blueberry juice and 50 % apple nectar, 30 % cranberry nectar and 40 % blueberry nectar have been made with minimum of 11.2 °Bx, 7.5 °Bx and 10 °Bx.

Compositions of 100 % juice (per 1 L) are: for apple juice: concentrated fruit juice (70±0.5 °Bx) 168 g, sugar 0 g, citric acid 0 g, water 881 g; and for 50 % apple nectar is: concentrated fruit juice 84 g, sugar 59 g, citric acid 3 g, water 927 g. Untreated samples were denoted A1.0 and A2.0 and ultrasound treated one as A1.1–A1.16 and A2.1–A2.16 (Table 1).

Compositions of 100 % juice (per 1 L) are: for cranberry juice: concentrated fruit juice (46±1 °Bx) 167 g, sugar 0 g, citric acid 0 g, water 833 g; and for 30 % cranberry nectar is: concentrated fruit juice 52 g, sugar 54 g, citric acid 3 g, water 960 g. Untreated samples were denoted A1.0 and A2.0 and ultrasound treated one as B1.1–B1.16 and B2.1–B2.16 (Table 1).

Compositions of 100 % juice (per 1 L) are: for blueberry juice: concentrated fruit juice (65±0.5 °Bx) 160 g, sugar 0 g, citric acid 0 g, water 880 g; and for 40 % blueberry nectar is: concentrated fruit juice 64 g, sugar 62 g, citric acid 3 g, water 914 g. Untreated samples were denoted A1.0 and A2.0 and ultrasound treated one as C1.1–C1.16 and C2.1–C2.16 (Table 1).

For each sample type, weighted amounts of fruit concentrate and water (for juices) and fruit concentrate, water, sugar and citric acid (for nectars) were mixed in bottle and used for ultrasound and pasteurized treatment.

Experimental methodology

In this study, experiment was designed in STATGRAPHICS Centurion (StatPoint technologies, Inc, Warrenton 20186, USA) software. Experiment consists of 16 experimental trials (Table 1). The independent variables were amplitude - X_1 (μm), temperature - X_2 (°C) and treatment time - X_3 (min). The operating variables were considered at three levels namely, low (−1), central (0) and high (1). Experiments were organized in a factorial design (including factorial points, axial points and centre point) and the remaining

Table 1 Experimental design of ultrasound treatment (HPU), denotation of samples of juices and nectars, power calculations (P) of ultrasound treatment and acoustic intensity (AI) and acoustic density (δ) calculations. Pasteurization of apple juice (A1), apple nectar (A2), cranberry juice (B1), cranberry nectar (B2), blueberry juice (C1) and blueberry nectar (C2) was done at 80 °C for 2 min

a) Apple juice and nectars

Amplitude (μm)	Temperature (°C)	Treatment time (min)	Sample of apple juice (100 %)	P (W)	AI ($\mathrm{W\,cm^{-2}}$)	δ ($\mathrm{W\,cm^{-3}}$)	Sample of apple nectar (50 %)	P (W)	AI ($\mathrm{W\,cm^{-2}}$)	δ ($\mathrm{W\,cm^{-3}}$)
90	60	6	A1.1	56	48.15	0.61	A2.1	56	46.58	0.59
60	60	9	A1.2	42	46.58	0.59	A2.2	42	47.36	0.60
90	40	3	A1.3	62	32.37	0.41	A2.3	60	31.58	0.40
90	40	6	A1.4	62	31.58	0.40	A2.4	59	31.58	0.40
120	40	6	A1.5	79	32.37	0.41	A2.5	76	32.37	0.41
120	20	3	A1.6	65	16.58	0.21	A2.6	72	17.37	0.22
120	60	3	A1.7	74	46.58	0.59	A2.7	71	46.58	0.59
90	40	6	A1.8	61	31.58	0.40	A2.8	62	32.37	0.41
60	60	3	A1.9	43	48.15	0.61	A2.9	44	48.15	0.61
60	40	6	A1.10	46	30.79	0.39	A2.10	47	31.58	0.40
120	20	9	A1.11	67	16.58	0.21	A2.11	70	16.58	0.21
60	20	3	A1.12	50	16.58	0.21	A2.12	52	16.58	0.21
90	20	6	A1.13	62	16.58	0.21	A2.13	61	16.58	0.21
120	60	9	A1.14	70	47.36	0.60	A2.14	73	46.58	0.59
90	40	9	A1.15	61	30.79	0.39	A2.15	62	31.58	0.40
60	20	9	A1.16	63	16.58	0.21	A2.16	48	16.58	0.21

b) Cranberry juice and nectars

Amplitude (μm)	Temperature (°C)	Treatment time (min)	Sample of cranberry juice (100 %)	P (W)	AI ($\mathrm{W\,cm^{-2}}$)	δ ($\mathrm{W\,cm^{-3}}$)	Sample of cranberry nectar (30 %)	P (W)	AI ($\mathrm{W\,cm^{-2}}$)	δ ($\mathrm{W\,cm^{-3}}$)
90	60	6	B1.1	59	48.15	0.61	B2.1	43	46.58	0.59
60	60	9	B1.2	44	47.36	0.60	B2.2	45	47.36	0.60
90	40	3	B1.3	63	32.37	0.41	B2.3	64	32.37	0.41
90	40	6	B1.4	62	30.79	0.39	B2.4	65	31.58	0.40
120	40	6	B1.5	81	31.58	0.40	B2.5	82	31.58	0.40
120	20	3	B1.6	48	15.79	0.20	B2.6	76	15.00	0.19
120	60	3	B1.7	74	47.36	0.60	B2.7	76	46.58	0.59
90	40	6	B1.8	65	31.58	0.40	B2.8	65	31.58	0.40
60	60	3	B1.9	43	47.36	0.60	B2.9	56	46.58	0.59
60	40	6	B1.10	49	30.00	0.38	B2.10	48	31.58	0.40
120	20	9	B1.11	63	15.79	0.20	B2.11	63	14.21	0.18
60	20	3	B1.12	50	15.79	0.20	B2.12	51	15.79	0.20
90	20	6	B1.13	64	16.58	0.21	B2.13	53	15.79	0.20
120	60	9	B1.14	75	47.36	0.60	B2.14	75	46.58	0.59
90	40	9	B1.15	63	32.37	0.41	B2.15	65	32.37	0.41

Table 1 (continued)

60	20	9	B1.16	48	15.00	0.19	B2.16	50	15.00	0.19
c) Blueberry juice and nectars										
Amplitude (μm)	Temperature (°C)	Treatment time (min)	Sample of blueberry juice (100 %)	P (W)	AI ($W cm^{-2}$)	δ ($W cm^{-3}$)	Sample of blueberry nectar (40 %)	P (W)	AI ($W cm^{-2}$)	δ ($W cm^{-3}$)
90	60	6	C1.1	56	47.36	0.60	C2.1	59	47.36	0.60
60	60	9	C1.2	45	45.79	0.58	C2.2	45	46.58	0.59
90	40	3	C1.3	63	31.58	0.40	C2.3	66	31.58	0.40
90	40	6	C1.4	63	30.79	0.39	C2.4	65	32.37	0.41
120	40	6	C1.5	81	31.58	0.40	C2.5	82	31.58	0.40
120	20	3	C1.6	64	14.21	0.18	C2.6	48	15.00	0.19
120	60	3	C1.7	74	47.36	0.60	C2.7	75	47.36	0.60
90	40	6	C1.8	63	32.37	0.41	C2.8	48	31.58	0.40
60	60	3	C1.9	45	47.36	0.60	C2.9	45	47.36	0.60
60	40	6	C1.10	49	31.58	0.40	C2.10	49	31.58	0.40
120	20	9	C1.11	53	14.21	0.18	C2.11	54	15.00	0.19
60	20	3	C1.12	49	14.21	0.18	C2.12	48	15.79	0.20
90	20	6	C1.13	64	15.00	0.19	C2.13	52	15.00	0.19
120	60	9	C1.14	75	47.36	0.60	C2.14	74	46.58	0.59
90	40	9	C1.15	61	31.58	0.40	C2.15	63	30.00	0.38
60	20	9	C1.16	49	15.00	0.19	C2.16	49	15.79	0.20

[a] Untreated samples were denoted A1.0 and A2.0 and ultrasound treated one as A1.1–A1.16 (apple juice) and A2.1–A2.16 (apple nectar)

[b] Untreated samples were denoted B1.0 and B2.0 and ultrasound treated one as B1.1–B1.16 (cranberry juice) and B2.1–B2.16 (cranberry nectar)

[c] Untreated samples were denoted C1.0 and C2.0 and ultrasound treated one as C1.1–C1.16 (blueberry juice) and C2.1–C2.16 (blueberry nectar)

involving the replication of the central point to get good estimate of experimental error. Repetition experiments were carried out after other experiments followed by order of runs designed by program. The designs were based on central composite design (*CCD*), face centered design characteristic with two centerpoints (Montgomery 2001; Myers and Montgomery 2002). The total number of experiments of the designs (N) can be calculated as follows,

$$\mathrm{N} = \mathrm{N_i} + \mathrm{N_o} + \mathrm{N_j} \tag{1}$$

Where $N_i=2^n$ is the number of experiments ($2^3=8$), N_o is the number of centre points and $N_j=2\times n$ ($2\times3=6$), is the number of star points (=2).

Design matrix for the experiment and the regression model proposed for response was given below:

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_{ii} X_i^2 + \sum_{i\langle j}^{n} \beta_{ij} X_i X_j \tag{2}$$

where β_0 is the value of the fixed response at the central point of the experiment which is the point (0, 0, 0); β_i, β_{ii} and β_{ij} are the linear, quadratic and cross-products coefficients, respectively. While demonstrating the significant effects 3-dimensional fitted surfaces were drawn (Kuehl 2000; Khuri and Cornell 1996). The model was fitted by multiple linear regressions (MLR). Calculations were done at 95 % of confidence level. Analysis of variance (ANOVA) was carried out to determine any significant differences ($p<0.05$) among the applied treatments. Analysis was carried out to assess whether the different treatments (pasteurisation or ultrasound treatment) conducted to statistically changes compared to untreated samples. The values not statistically different are accompanied by the letter (a) and the values statistically different with the letter (b). The level of significance was 0.05 ($\alpha=0.05$).

Ultrasound treatments (HPU)

Apple, cranberry, blueberry juice or nectar sample (100 mL) was placed in a round bottom glass (200 mL), which served as the treatment chamber. An ultrasonic processor (S-4000, Misonix Sonicators, Newtown, Connecticut, USA), set at 600 W, 20 kHz, 12–260 μm with a 12.7 mm diameter probe, was introduced into the vessel. Ultrasonication was carried out with 60, 90 and 120 μm amplitude. Juice and nectar samples were treated by ultrasounds for 3, 6 and 9 min at 20 °C. In the case of thermosonication before ultrasonic treatment the samples were heated at 40 °C and 60 °C. Overheating of the samples, during ultrasound treatment, was prevented by ice-water cooling of the treatment chamber during sonication. For this study, 16 samples of juices and 16 samples of nectars for each fruit were ultrasonically treated (Table 1a, b and c).

Determination of acoustic power

The most widely accepted method for determining the power from an acoustic horn into an aqueous solution is the calorimetric technique described by Margulis and Maltsev (2003). This method involves taking a known volume of water and applying ultrasound (for ca. 3 min) while monitoring the change in temperature with time for various ultrasonic amplitudes. The ultrasonic power can be readily determined from the following equation:

$$P = m \times C_p \times \left(\frac{\partial T}{\partial t}\right)_{t=0} \tag{3}$$

Acoustic intensity has been calculated following equation:

$$\mathrm{AI} = \mathrm{P/A} \tag{4}$$

Acoustic density has been calculated according equation:

$$\delta = \mathrm{P/V} \tag{5}$$

where P is the ultrasonic power (W), m is the mass of the sample (kg), c_p is the specific heat capacity of apple, cranberry and blueberry juices and nectars ($\mathrm{kJ\,kg^{-1}\,°C^{-1}}$), AI—is the ultrasonic intensity ($\mathrm{W\,cm^{-2}}$), A is the surface area of probe ($\mathrm{cm^2}$), δ - acoustic density ($\mathrm{W\,cm^{-3}}$) and V-volume of sample ($\mathrm{cm^3}$).

Pasteurisation procedure

For comparison of the achieved effects of ultrasound on the investigated parameters, parallel pasteurization process was carried out. Pasteurization of samples was carried out on the heater with a magnetic mixer (IKA RTC Basic, Ika-Werke GmbH and Co.KG, Janke and Kunkel, Staufen 79 219, Germany) where the samples (100 mL) were in glass covered with aluminum foil over hot water bath at a temperature of 80 °C for 2 min (Table 1a, b and c). Pasteurized samples have been denoted A1.P (apple juice) and A2.P (apple nectar), B1.P (cranberry juice) and B2.P (cranberry nectar), C1.P (blueberry juice) and C2.P (blueberry nectar).

Determination of rheological properties of model systems

Torque measurements were carried out on the model systems using a Rheometric Viscometer (Model RM 180, Rheometric Scientific, Inc., Piscataway, USA) with the spindle (no. 3; $Ø=14$ mm; $l=21$ cm). Shear stress against the increasing shear rates from the lowest value of 0 $\mathrm{s^{-1}}$ to 1,290 $\mathrm{s^{-1}}$, as well as downwards, was applied. The volume of the beaker was 36 mL. The samples were kept in a thermostatically controlled water bath for about 15 min before measurements, in order to attain the desirable temperature of 25 ^{0}C. Measurements were done in triplicates for each sample. The shear rate versus shear stress was interpreted using the Rheometric computer program. The values for n and k were obtained from plots of log shear stress versus log shear rate, according to the power law equation:

$$\log \tau = \log k + n \log \gamma \tag{6}$$

where τ is the shear stress (Pa); γ is the shear rate (s^{-1}); n is the flow behavior index, and k is the consistency coefficient (Pa s^n).

Apparent viscosity (η_{app}) was calculated at 1,290 s^{-1} using Newtonian law, in addition to linear least square method for regression analysis.

$$\tau = \eta_{app}\gamma \tag{7}$$

Results and discussion

Acoustic intensity applied in sonication of apple juices and nectars varied from 16.58 to 48.15 $W cm^{-2}$ (Table 1a). The highest intensity of ultrasound treatment (maximum power per unit area of embedded probes) was determined in samples treated at 60 °C, and lowest in samples treated at 20 °C. The highest intensity of ultrasound treatment was found for sample of cranberry juice B1.1 (48.15 $W cm^{-2}$), and lowest in treatment B2.11 (14.21 $W cm^{-2}$) (Table 1b). The highest intensity of ultrasound treatment was found for blueberry samples C1.1, C2.1, C1.7, C2.7, C1.14 and C2.9 (47.36 $W cm^{-2}$) also at temperatures of 60 °C, and lowest in treatment C1.6, C1.11 and C1.12 (14.21 $W cm^{-2}$) (Table 1c).

Influence of ultrasound on rheological properties of ultrasound treated apple, cranberry and blueberry juice and nectar

Using design of experiment, several statistical analyses could be conducted and also test the influence of combined and individual effect of certain factors (amplitude (A), treatment time (B) and temperature (C)) on output parameters. Using just results of analysis variance, it is not possible to determine the combined impact of several factors, while in simple versions of the experiment ("one by one factor") only influence of one factor is tested. Also, with statistical design of processing parameters one can analysis quadratic interaction (AA, AB, AC, BB, BC, CC) of investigated parameters amplitude level (μm) = A, sonication time (min) = B and temperature (°C) = C on response results (output value).

Rheological properties are expressed with consistency coefficient (k) and flow behavior indices (n), and are adequately described with Ostwald de Wale's power law. After 20 kHz ultrasound treatment of fruit juice there have been neither significant changes ($p>0.05$) in apparent viscosity (Tables 2, 3 and 4) regardless fruit juice type or ultrasound treatment. From Tables 2, 3 and 4 it was found that all samples of untreated, pasteurized and ultrasonically treated apple, cranberry and blueberry juices and nectars shows

Table 2 Rheological parameters of untreated, pasteurized and ultrasound treated apple juice and nectar (A.1 and A.2). Untreated samples were denoted A1.0 and A2.0 and ultrasound treated one as A1.1–A1.16 (apple juice) and A2.1–A2.16 (apple nectar).; pasteurized samples have been denoted A.1.P (apple juice) and A.2.P (apple nectar)

Sample	Apparent viscosity μ (mPas)[a]	Consistency coefficient k (Pa s^n) × 10^{-5}	Flow behaviour index n	Regression coefficient R^2
A1.0	6	2.692	1.754	0.999
A1.P	5	1.230	1.851	0.998
A1.1	5	2.265	1.767	0.999
A1.2	5	1.667	1.808	0.994
A1.3	5	1.330	1.841	0.999
A1.4	6	1.629	1.813	0.998
A1.5	6	3.055	1.727	0.998
A1.6	6	3.350	1.719	0.997
A1.7	5	1.253	1.851	0.998
A1.8	6	1.959	1.791	0.999
A1.9	5	1.030	1.878	0.998
A1.10	5	1.746	1.808	0.997
A1.11	6	1.611	1.818	0.996
A1.12	6	3.846	1.703	0.986
A1.13	6	2.805	1.747	0.996
A1.14	6	2.158	1.780	0.998
A1.15	6	2.323	1.773	0.999
A1.16	6	2.864	1.744	0.997
A2.0	6	2.307	1.767	0.999
A2.P	5	1.315	1.842	0.998
A2.1	5	1.592	1.815	0.998
A2.2	6	1.694	1.812	0.997
A2.3	5	1.306	1.840	0.999
A2.4	5	1.390	1.832	0.998
A2.5	6	1.879	1.797	0.998
A2.6	5	3.141	1.722	0.998
A2.7	6	1.112	1.867	0.999
A2.8	6	1.250	1.855	0.994
A2.9	5	2.818	1.733	0.998
A2.10	6	2.333	1.763	0.996
A2.11	6	1.180	1.860	0.994
A2.12	5	1.778	1.799	0.998
A2.13	6	1.028	1.878	0.998
A2.14	5	1.552	1.817	0.998
A2.15	6	1.932	1.792	0.996
A2.16	5	1.285	1.846	0.998

[a] at 1290 s^{-1}

Table 3 Rheological parameters of untreated, pasteurized and ultrasound treated cranberry juice and nectar (B.1 and B.2). Untreated samples were denoted B1.0 and B2.0 and ultrasound treated one as B1.1–B1.16 (cranberry juice) and B2.1–B2.16 (cranberry nectar).; pasteurized samples have been denoted B.1.P (cranberry juice) and B.2.P (cranberry nectar)

Sample	Apparent viscosity μ (mPas)[a]	Consistency coefficient k (Pa s^n) × 10^{-5}	Flow behaviour index n	Regression coefficient R^2
B1.0	6	2.042	1.784	0.998
B1.P	5	1.186	1.854	0.999
B1.1	5	1.318	1.840	0.999
B1.2	5	2.547	1.743	0.995
B1.3	5	1.042	1.865	0.998
B1.4	5	1.698	1.798	0.999
B1.5	5	2.371	1.760	0.999
B1.6	5	1.318	1.834	0.997
B1.7	5	2.123	1.766	0.999
B1.8	5	1.416	1.831	0.999
B1.9	5	1.393	1.827	0.999
B1.10	5	1.327	1.836	0.996
B1.11	5	1.626	1.806	0.999
B1.12	5	1.442	1.822	0.998
B1.13	5	1.387	1.831	0.996
B1.14	5	0.964	1.878	0.998
B1.15	5	1.021	1.874	0.997
B1.16	5	1.560	1.815	0.998
B2.0	6	1.648	1.805	0.999
B2.P	5	1.667	1.801	0.997
B2.1	5	1.510	1.818	0.999
B2.2	5	1.091	1.862	0.997
B2.3	6	1.091	1.862	0.997
B2.4	5	1.600	1.810	0.997
B2.5	5	2.163	1.769	0.999
B2.6	5	1.941	1.785	0.999
B2.7	5	1.778	1.791	0.999
B2.8	5	1.096	1.862	0.999
B2.9	5	1.309	1.830	0.995
B2.10	5	1.122	1.863	0.998
B2.11	5	1.963	1.784	0.999
B2.12	5	2.198	1.770	0.998
B2.13	5	1.236	1.846	0.999
B2.14	5	0.973	1.869	0.998
B2.15	5	0.914	1.887	0.997
B2.16	5	1.175	1.855	0.999

[a] at 1290 s^{-1}

Table 4 Rheological parameters of untreated, pasteurised and ultrasound treated blueberry juice and nectar (C.1 and C.2). Untreated samples were denoted C1.0 and C2.0 and ultrasound treated one as C1.1–C1.16 (blueberry juice) and C2.1–C2.16 (blueberry nectar).; pasteurized samples have been denoted C.1.P (blueberry juice) and C.2.P (blueberry nectar)

Sample	Apparent viscosity μ (mPas)[a]	Consistency coefficient k (Pa s^n) × 10^{-5}	Flow behaviour index n	Regression coefficient R^2
C1.0	6	2.488	1.760	0.999
C1.P	5	1.618	1.805	0.998
C1.1	5	2.050	1.776	0.997
C1.2	6	2.883	1.739	0.998
C1.3	5	2.564	1.748	0.995
C1.4	6	2.289	1.767	0.999
C1.5	6	2.919	1.733	0.999
C1.6	6	2.817	1.738	0.998
C1.7	5	1.923	1.782	0.997
C1.8	5	1.510	1.823	0.999
C1.9	5	1.109	1.858	0.997
C1.10	5	2.002	1.783	0.996
C1.11	6	2.204	1.772	0.998
C1.12	6	2.764	1.744	0.998
C1.13	6	1.803	1.802	0.999
C1.14	6	2.330	1.764	0.998
C1.15	5	1.625	1.870	0.997
C1.16	6	1.285	1.850	0.999
C2.0	6	2.549	1.759	0.998
C2.P	5	2.432	1.756	0.999
C2.1	5	2.107	1.777	0.998
C2.2	6	2.178	1.775	0.997
C2.3	5	2.157	1.773	0.997
C2.4	6	1.688	1.810	0.997
C2.5	6	2.450	1.760	0.999
C2.6	6	2.994	1.735	0.998
C2.7	5	2.510	1.748	0.998
C2.8	5	2.541	1.750	0.998
C2.9	6	1.860	1.794	0.998
C2.10	5	2.778	1.734	0.997
C2.11	6	1.805	1.802	0.998
C2.12	6	2.156	1.779	0.998
C2.13	6	2.551	1.754	0.999
C2.14	5	2.594	1.744	0.998
C2.15	5	2.061	1.780	0.994
C2.16	6	2.496	1.756	0.998

[a] at 1290 s^{-1}

non-Newtonian dilatant fluid characteristics ($n>1$). The consistency coefficient (k) in treated samples of apple, cranberry and blueberry juices and nectars, decreases and increases depending on the applied ultrasound treatment. Thus, the largest increase in consistency coefficient (3.846×10^{-5} Pa s^n) in apple juice samples was determined after ultrasonic treatment A1.12 (amplitude 60 μm, 3 min treatment time and the sample temperature of 20 °C), and decrease in consistency coefficient (1.03×10^{-5} Pa s^n) for sample A1.9 (amplitude 60 %, 3 min treatment time and the sample temperature of 60 °C). For samples of apple nectar largest increase in consistency coefficient (3.141×10^{-5} Pa s^n) was determined after ultrasonic treatment of A2.6 (amplitude 120 μm, 3 min treatment time and the sample temperature of 20 °C), and decrease in consistency coefficient (1.028×10^{-5} Pa s^n) for sample A2.13 (amplitude 90 μm, 6 min treatment time and the sample temperature of 20 °C). The effect of temperature on viscosity of juice is different for distinct temperature range. The viscosity of juices is considerably affected by temperature below 360 K. At constant concentrations from 15 to 40 °Brix between temperatures 303 and 360 K, the viscosity of juices changes by factor of 4.5. The temperature effect on viscosity strongly depends also on the concentration of juice. At low concentrations (below 30 °Brix) the rate of viscosity changes is small; the effect of concentration on the viscosity of juices strongly depends on temperature and concentration. At $x<20$ °Brix, the viscosity is little affected by concentration, while at high concentrations the effect of concentration stronger (Magerramov et al. 2007). However, in ultrasound treatment with elevated temperature there is no evident influence of temperature on consistency coefficient (k) where somewhere it increases at 20 °C or decreases at 60 °C. But there is evident decrease in consistency coefficient (k) for pasteurized samples, where is decreased when compared to untreated samples.

The largest increase in consistency coefficient (2.547×10^{-5} Pa s^n) of cranberry juice was found in sample B1.2 (amplitude 60 μm, 9 min treatment time and the sample temperature of 60 °C), and decrease in consistency coefficient (0.964×10^{-5} Pa s^n) for sample B1.14 (120 um amplitude / time of 9 min and the sample temperature of 60 °C). For samples of cranberry nectar largest increase in consistency coefficient (2.198×10^{-5} Pa s^n) was determined after ultrasonic treatment of B2.12 (amplitude 60 μm, 3 min treatment time and the sample temperature of 20 °C), and decrease in consistency coefficient (0.914×10^{-5} Pa s^n) for sample B2.15 (amplitude 90 μm, 9 min treatment time and the sample temperature of 40 °C). From Table 4 the largest increase in consistency coefficient (2.919×10^{-5} Pa s^n) is observed for blueberry juice after ultrasonic treatment of C1.5 (amplitude 120 μm, 6 min treatment time and the sample temperature of 40 °C), and decrease in consistency coefficient (1.109×10^{-5} Pa s^n) for sample C1.9 (amplitude 60 μm, 3 min treatment time and the sample temperature of 60 °C). For samples of blueberry nectar largest increase in consistency coefficient (2.994×10^{-5} Pa s^n) was determined after ultrasonic treatment of C2.6 (amplitude 120 μm, 3 min treatment time and the sample temperature of 20 °C), and decrease in consistency coefficient (1.688×10^{-5} Pa s^n) for sample C2.4 sample (amplitude 90 μm, 6 min treatment time and the sample temperature of 40 °C). These results stem from differences in the composition of fruit components (concentrate) from which samples were prepared, but also the fact the differences between the composition of juices and nectars. An important influence on the rheological properties of the nectar is sucrose. Because of different chemical composition and the presence of sucrose and other sugar systems show differences in behavior (Dumay et al. 1994). The molecular movement in a solution is affected by the amount of molecules present and their interaction with water molecules. The effect of concentration on the viscosity is an important factor in food processing. Benitez et al. (2009) showed that specific viscosity of a colloidal dispersion of solids in syrups is increased by increasing particle-sugar interactions and by lowering the water activity of syrups. The sugar composition and processing procedures for juices and nectars are likely factors that can explain the differences in rheological properties. Whereas glucose and fructose are present in almost equal proportion in the two berries concentrate, apple concentrate contains more sucrose and total sugars than berry juices and nectar. Solute type, size, shape, and state of hydration all have an effect on viscosity (Fennema 1996). Sucrose, with a higher molecular weight than either glucose or fructose, has a higher viscosity for a solution of the same concentration. Suspended particles remaining in the juice after depectinization and filtration processes may influence the viscosity of the juices (Hernandez et al. 1995).

General properties of ultrasound, which is used in food processing is the fact that high power ultrasound can cause changes in some properties (chemical, functional, physical, etc.) which may be interesting as a technological advantage (Režek Jambrak et al. 2008).

Results of rheological parameters (*K* and *n*) were statistically processed and analyzed using statistical software in design of experiment. According to the results of analysis of variance, Table 5 shows that the interaction of treatment time and temperature of sample (BC) and temperature (C) of sample of apple juice had statistically significant ($p<0.05$) effect on flow behavior index (*n*) for ultrasound treated apple juice. There is no significant ($p>0.05$) effect of other investigated parameters and their interaction on flow behavior index (*n*). Interaction of treatment time and temperature of sample (BC) has statistically significant ($p<0.05$) effect on the flow behavior index (n) for blueberry nectar. Also, there is statistically significant ($p<0.05$) effect of temperature (C) of sample on consistency coefficient (*k*) for ultrasound treated apple juice. There is no significant ($p>0.05$) effect of other investigated parameters and their interaction on consistency coefficient (*k*). The linear relationship between dependent and independent variables can be evaluated by plotting the residues and verifying if the errors are randomly distributed. The errors independence can be checked using the Durbin–Watson statistics (a value near to 2 will indicate that the errors are independent). On contrary, for blueberry juice values of Durbin–Watson

Table 5 Analysis of variance (ANOVA) for ultrasound parameters (amplitude: A, treatment time: B, and temperature: C) and their quadratic interactions (AA, AB, AC, BB, BC, CC) for response values (rheological parameters: flow behaviour index (n), and consistency coefficient (k (Pa s^n) × 10^{-5}))

Parameters	Sample	*F*-Value		*P*-Value	
		n	*k*	*n*	*k*
A:amplitude	apple juice	0.15	0.02	0.7098	0.8949
	apple nectar	0.56	0.29	0.481	0.6081
	cranberry juice	0	0.01	0.9945	0.9376
	cranberry nectar	2.5	2.12	0.1646	0.1957
	blueberry juice	1.75	1.73	0.2345	0.2361
	blueberry nectar	0.35	0.55	0.5758	0.4866
AA	apple juice	0.18	0.3	0.6897	0.6026
	apple nectar	2.19	2.13	0.1896	0.1951
	cranberry juice	2.49	2.48	0.1657	0.1661
	cranberry nectar	2.22	2.3	0.1871	0.18
	blueberry juice	1.26	1.19	0.3047	0.3177
	blueberry nectar	1.23	1.59	0.3098	0.2544
AB	apple juice	0.29	0.08	0.6081	0.7925
	apple nectar	0.08	0	0.7815	0.9575
	cranberry juice	2.01	2.12	0.2061	0.1953
	cranberry nectar	0.15	0.15	0.7108	0.7116
	blueberry juice	0.05	0.12	0.8298	0.7433
	blueberry nectar	2.09	2.73	0.1986	0.1499
AC	apple juice	1.89	1.92	0.2181	0.2153
	apple nectar	2.38	3.24	0.174	0.1221
	cranberry juice	0.33	0.3	0.5862	0.605
	cranberry nectar	0.05	0.02	0.8233	0.8839
	blueberry juice	0.07	0.24	0.7969	0.6438
	blueberry nectar	1.22	0.74	0.3121	0.4225
B: treatment time	apple juice	0.34	0.01	0.5795	0.9285
	apple nectar	1.28	1.69	0.3003	0.2408
	cranberry juice	0	0.06	0.9889	0.8142
	cranberry nectar	3.63	2.78	0.1055	0.1467
	blueberry juice	0.78	0.27	0.4112	0.6214
	blueberry nectar	0.12	0.21	0.7392	0.6653
BB	apple juice	1.69	0.87	0.2407	0.3868
	apple nectar	0.05	0.03	0.8354	0.8763
	cranberry juice	1.17	1	0.3208	0.3555
	cranberry nectar	1.26	0.94	0.3043	0.3692
	blueberry juice	0.55	0	0.4858	0.9534
	blueberry nectar	0.57	0.83	0.4795	0.3972
BC	apple juice	7.11	5.75	***0.0372***	0.0535
	apple nectar	1.42	1.05	0.2787	0.3448
	cranberry juice	0.26	0.09	0.6281	0.7773
	cranberry nectar	0.06	0	0.8089	0.9857
	blueberry juice	4.88	8.55	0.0691	***0.0265***
	blueberry nectar	0.83	1.37	0.3963	0.2858
C:temperature	apple juice	8.97	9.42	***0.0242***	***0.0219***
	apple nectar	0.17	0.03	0.6915	0.8597
	cranberry juice	0.15	0.39	0.7092	0.5573
	cranberry nectar	1.28	1.97	0.3014	0.2104

Table 5 (continued)

Parameters	Sample	*F*-Value		*P*-Value	
		n	*k*	*n*	*k*
	blueberry juice	0.01	0.13	0.9352	0.7355
	blueberry nectar	0.02	0.4	0.8817	0.5515
CC	apple juice	0.77	0.8	0.4144	0.4044
	apple nectar	0.73	0.43	0.4268	0.534
	cranberry juice	0.03	0	0.861	0.9916
	cranberry nectar	0.6	0.22	0.4681	0.6553
	blueberry juice	0	0.35	0.9984	0.5773
	blueberry nectar	0.01	0	0.9436	0.9734
	Sample	Parameter			
		n	*k*		
R-squared (%)	apple juice	77.5799	75.8712		
	apple nectar	58.9645	59.6962		
	cranberry juice	49.4118	49.2993		
	cranberry nectar	65.9794	63.301		
	blueberry juice	60.0234	67.2713		
	blueberry nectar	50.2378	56.9137		
R-squared (adjusted for d.f.) (%)	apple juice	43.9498	39.678		
	apple nectar	0	0		
	cranberry juice	0	0		
	cranberry nectar	14.9486	8.25257		
	blueberry juice	0.0585536	18.1782		
	blueberry nectar	0	0		
Standard error of estimation	apple juice	0.0372663	0.628697		
	apple nectar	0.0463141	0.610369		
	cranberry juice	0.0436517	0.515094		
	cranberry nectar	0.0363637	0.417699		
	blueberry juice	0.0444093	0.516522		
	blueberry nectar	0.025664	0.377576		
Mean absolute error	apple juice	0.0204733	0.342815		
	apple nectar	0.0246638	0.325978		
	cranberry juice	0.0228017	0.263278		
	cranberry nectar	0.01965	0.23125		
	blueberry juice	0.02291	0.289413		
	blueberry nectar	0.0125994	0.192751		
Durbin-Watson statistic	apple juice	2.03203 (*P*=0.4276)	2.04001 (*P*=0.4351)		
	apple nectar	1.73234 (*P*=0.1845)	1.65248 (*P*=0.1374)		
	cranberry juice	2.11507 (*P*=0.5061)	2.17979 (*P*=0.5673)		
	cranberry nectar	1.83604 (*P*=0.2581)	1.85274 (*P*=0.2711)		
	blueberry juice	***1.03492 (P=0.0033)***	***1.08013 (P=0.0049)***		
	blueberry nectar	0.18597 (*P*=0.2767)	1.79349 (*P*=0.2262)		
Lag 1 residual autocorrelation	apple juice	−0.0952045	−0.0760406		
	apple nectar	0.0765804	0.136151		
	cranberry juice	−0.0849697	−0.114395		
	cranberry nectar	0.0490244	0.0312844		
	blueberry juice	0.470367	0.437182		
	blueberry nectar	0.0668961	0.0986123		

Bolded values represent statistical significance

Table 6 Optimisation polynoms, optimal values and optimal ultrasound parameters for rheological parameters, flow behaviour index (n) and consistency coefficient (k (Pas^n) × 10^{-5}) of analysed samples after ultrasound treatments

			Apple								Cranberry								Blueberry							
			Juice				Nectar				Juice				Nectar				Juice				Nectar			
Rheological parameters	flow behaviour index (n)	Polynomial fit	(apple juice) $n=1.48836+0.00250414A-0.0258474B+0.0120204C-0.0000106897A^{2}+0.0000791667AB-0.0000302083AC+0.00331992B^{2}-0.000585417BC-0.0000503017C^{2}$																							
			(apple nectar) $n=1.51307+0.00743793A+0.0315362B-0.00700457C-0.0000468774A^{2-}0.0000527778AB+0.0000420833AC-0.000687739B^{2-}0.000325BC+0.0000607759C^{2}$																							
			(cranberry juice) $n=1.77284+0.00643609A-0.0658399B-0.00140599C-0.0000471264A^{2}+0.000243056AB+0.0000147917AC+0.0032318B^{2}+0.00013125BC-0.0000122845C^{2}$																							
			(cranberry nectar) $n=1.56183+0.00619563A-0.0234069B+0.00334397C-0.0000370498A^{2}-0.0000555556AB+0.000005AC+0.00279502B^{2}+0.0000541667BC-0.0000433621C^{2}$																							
			(blueberry juice) $n=1.53258+0.00500329A-0.0033342B+0.00288437C-0.0000341034A^{2}+0.0000391667AB+0.00000704167AC+0.00225632B^{2-}0.000578333BC+1.42241E\text{-}7C^{2}$																							
			(blueberry nectar) $n=1.65647+0.00314023A-0.0225315B+0.00216052C-0.0000194828A^{2}+0.000145694AB-0.0000166875AC+0.00132395B^{2-}0.000138125BC+0.00000291379C^{2}$																							
		Optimal values	1.70315				1.74712				1.76844				1.75816				1.70623				1.73915			
		Optimal factors	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*
			A	60.0	120.0	60.0	A	60.0	120.0	60.0	A	60.0	120.0	60.0	A	60.0	120.0	120.0	A	60.0	120.0	120.0	A	60.0	120.0	120.0
			B	3.0	9.0	4.93	B	3.0	9.0	3.0	B	3.0	9.0	6.69	B	3.0	9.0	5.17	B	3.0	9.0	3.0	B	3.0	9.0	4.99
			C	20.0	60.0	20.00	C	20.0	60.0	44.88	C	20.0	60.0	60.0	C	20.0	60.0	20.0	C	20.0	60.0	20.0	C	20.0	60.0	59.93
	consistency coefficient (k) (Pas^n) × 10^{-5}	Polynomial fit	(apple juice) $k=8.70243-0.0580815A+0.181376B-0.199429C+0.00023636A^{2-}0.000679167AB+0.000513125AC-0.0401418B^{2}+0.00888125BC+0.00086806C^{2}$																							
			(apple nectar) $k=5.03569-0.0880105A-0.324659B+0.0874787C+0.000608966A^{2}+0.000133333AB-0.000647083AC+0.00678544B^{2}+0.0036875BC-0.000619828C^{2}$																							
			(cranberry juice) $k=1.93575-0.0752366A+0.737981B+0.0246572C+0.000555536A^{2-}0.00294861AB-0.000165625AC-0.0352797B^{2-}0.000897917BC+0.0000087069C^{2}$																							
			(cranberry nectar) $k=4.71232-0.0739653A+0.204159B-0.0297686C+0.000433659A^{2}+0.000636111AB-0.0000375AC-0.0277452B^{2-}0.0000458333BC+0.000301983C^{2}$																							
			(blueberry juice) $k=5.20279-0.052054A-0.29593B-0.00549194C+0.000385115A^{2-}0.000695833AB-0.000148125AC-0.00215517B^{2}+0.00890208BC-0.000468491C^{2}$																							
			(blueberry nectar) $k=4.06049-0.0486305A+0.3806B-0.0382475C+0.000325632A^{2-}0.00244861AB+0.000191458AC-0.0235479B^{2}+0.00260625BC+0.0000201724C^{2}$																							
		Optimal values	0.733223				0.743805				0.838016				0.706637				1.23239				1.71771			
		Optimal factors	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*	*Factor*	*Low*	*High*	*Optimal*
			A	60.0	120.0	62.05	A	60.0	120.0	81.88	A	60.0	120.0	78.78	A	60.0	120.0	81.05	A	60.0	120.0	79.63	A	60.0	120.0	68.18
			B	3.0	9.0	3.0	B	3.0	9.0	8.99	B	3.0	9.0	3.0	B	3.0	9.0	9.0	B	3.0	9.0	9.0	B	3.0	9.0	3.0
			C	20.0	60.0	60.0	C	20.0	60.0	20.0	C	20.0	60.0	20.02	C	20.0	60.0	55.00	C	20.0	60.0	20.00	C	20.0	60.0	60.0

A amplitude, *B* treatment time, *C* temperature, quadratic interactions (AA = A^2 , AB, AC, BB = B^2 , BC, CC = C^2)

statistics are statistically significant and around 1 which means that errors are dependent. The homogeneity of variances can be checked graphically by plotting the standardized residues against the estimated values. If the residues are spread more or less randomly around zero, the variance is constant.

Table 6 gives optimisation polynoms, optimal values and optimal ultrasound parameters for rheological parameters, flow

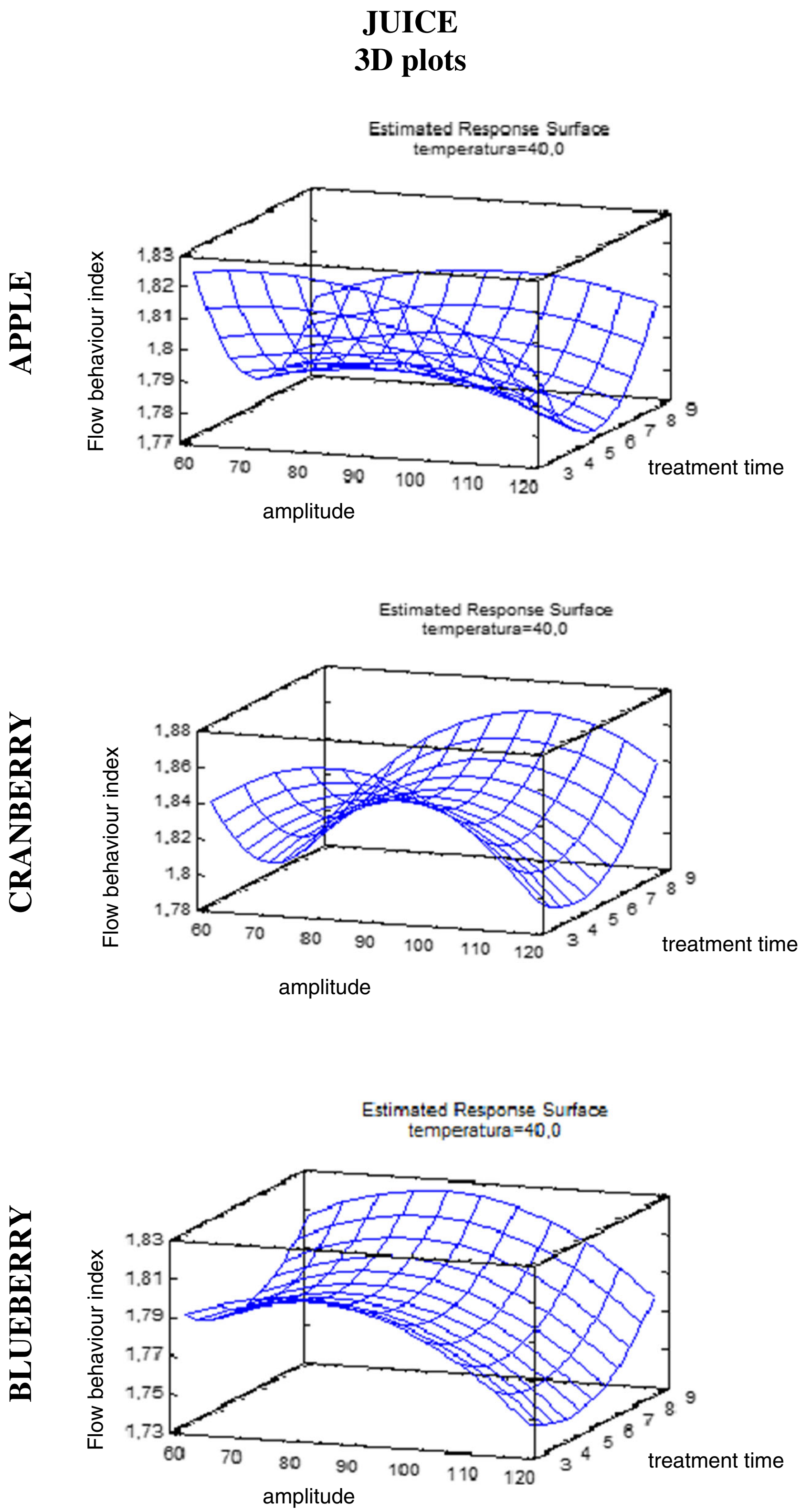
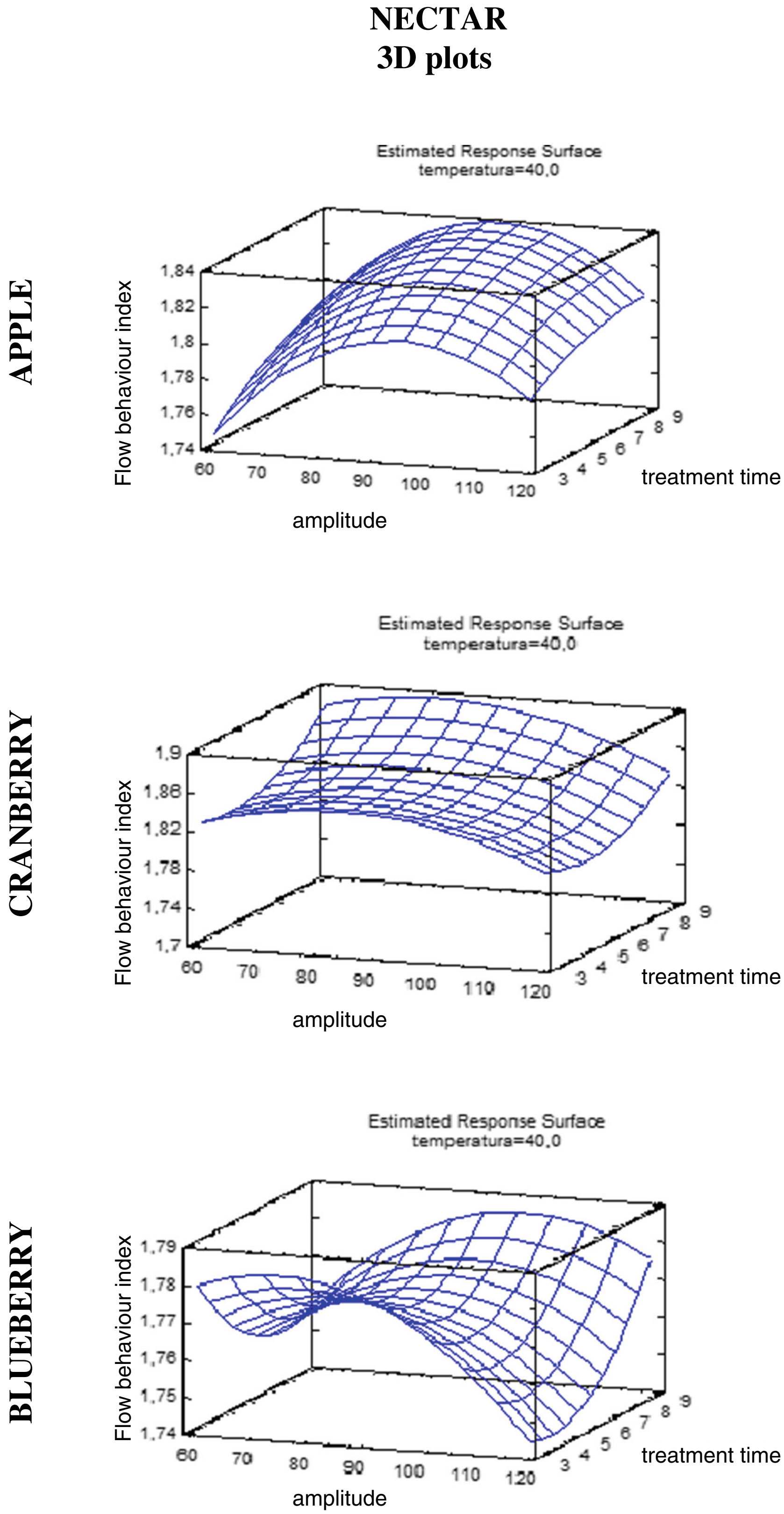


Fig. 1 Response surface 3D plots for rheological parameter flow behaviour indices (n) for fruit juices

behaviour index (n) and consistency coefficient (k (Pas^n) × 10^{-5}) of analysed samples (of apple, cranberry and blueberry juice and nectar) after ultrasound treatments. The effects of independent variables of ultrasound parameters processing time (3, 6, 9 min) on the rheological parameters were fitted to second order polynomial models. From the experimental data and

Fig. 2 Response surface 3D plots for rheological parameter flow behaviour indices (n) for fruit nectars



Eq. (2), the second-order response functions were expressed as a function of the independent variables as shown Table 6.

3D plots representing the linear and quadratic effects of the independent variables for the rheological parameters

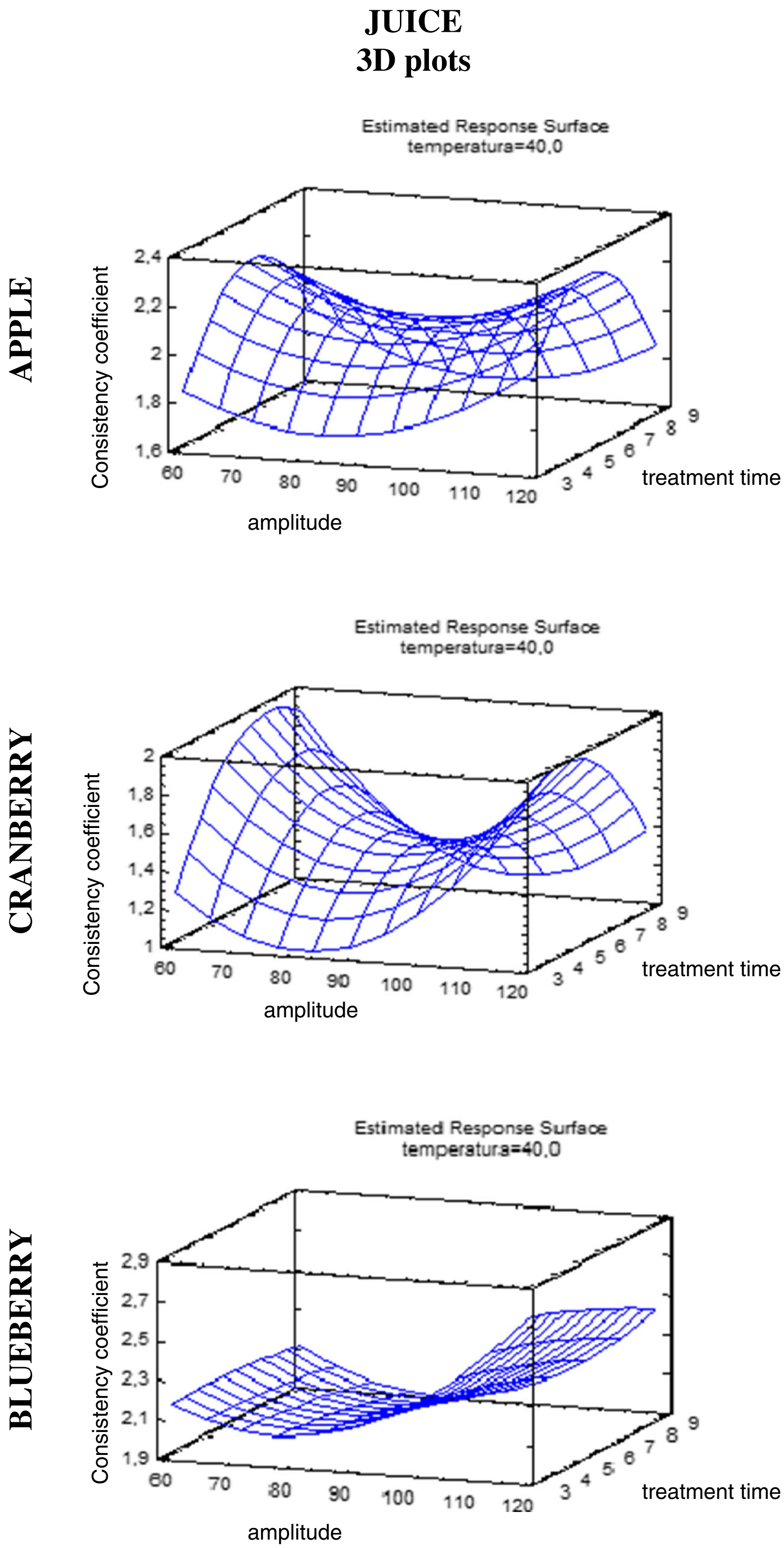
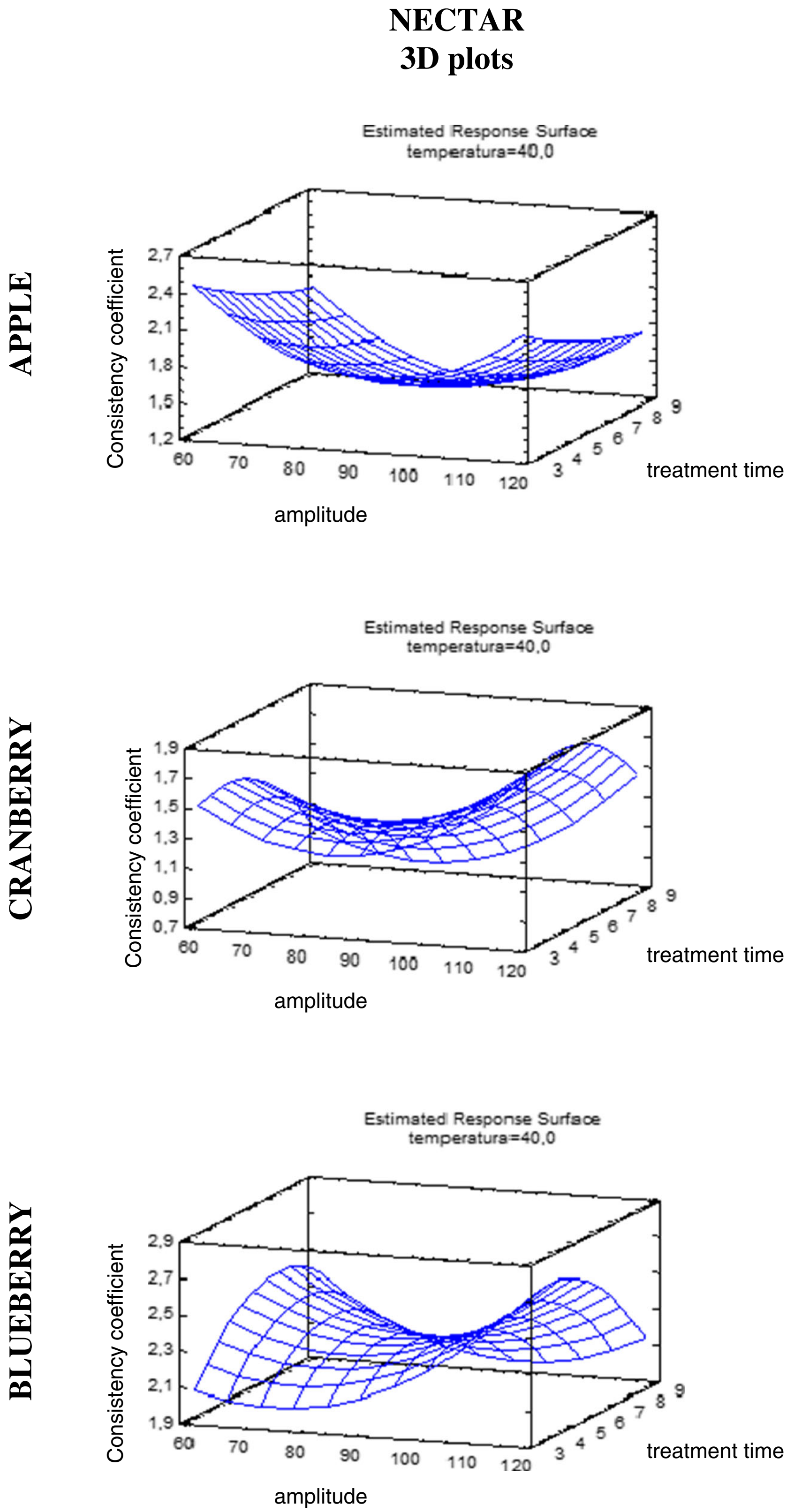


Fig. 3 Response surface 3D plots for rheological parameter consistency coefficient (k) for fruit juices

(n and k) are presented in Figs. 1, 2, 3 and 4. The predicted response models were found to fit well with the experimental data with low standard error and high regression coefficients. To confirm the adequacy of the fitted models, the normality assumption was satisfied as the residual plot approximated to a straight line for all responses.

Fig. 4 Response surface 3D for rheological parameter consistency coefficient (k) for fruit nectars



Response surface (3D) obtained on the basis of the polynomial optimization of Table 6, which was determined using the optimum parameters of amplitude, treatment time and temperature of ultrasonic treatment are given in Figs. 1, 2, 3 and 4. Plots are given at fixed temperature of 40 °C for each juice and nectar sample. Use of high power ultrasound has been employed in several applications of fruit processing. The effect of high-intensity ultrasound on the rheological and optical properties of high-methoxyl pectin dispersions was studied by Seshadri et al. (2003). A power law model was fitted to the flow curves of ultrasonically pre-treated pectin dispersions to determine both flow behavior index *n* and consistency coefficient *K*. With increased sonication power and application time, *n* increased from 0.6 to 0.97 indicating that the flow behaviour changed from viscoelastic to Newtonian (Seshadri et al. 2003).

Conclusion

The purpose of this investigation was to examine the influence of high power ultrasound on changes in rheological parameters (n and k) of ultrasound treated apple, cranberry, blueberry juice and nectar. It was found that all samples of untreated, pasteurized and ultrasonically treated apple, cranberry and blueberry juices and nectars shows non-Newtonian dilatant fluid characteristics ($n>1$). The consistency coefficient (*k*) in treated samples of apple, cranberry and blueberry juices and nectars, decreases and increases depending on the applied ultrasound treatment. The interaction of treatment time and temperature of sample (BC) and temperature (C) of sample of apple juice had statistically significant effect on flow behavior index (*n*) for ultrasound treated apple juice. There is no significant effect of other investigated parameters and their interaction on flow behavior index (*n*). Interaction of treatment time and temperature of sample (BC) has statistically significant effect on the flow behavior index (n) for blueberry nectar. Also, there is statistically significant effect of temperature (C) of sample on consistency coefficient (*k*) for ultrasound treated apple juice. There is no significant effect of other investigated parameters and their interaction on consistency coefficient (*k*). The use of novel non-thermal processing is well known. Several novel and interesting applications for improving the technological properties of food have emerged during the past few years. These technologies represent a rapid, efficient and reliable alternative to improve the quality of food, but it also has the potential to develop new product and improving their nutritive quality.

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