

Aroma Profile and Sensory Properties of Ultrasound-Treated Apple Juice and Nectar

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Summary

Ultrasonication is a nonthermal food processing method that is used in several applications (extraction, treatment before drying, freezing, inactivation of microorganisms, *etc.*) in ultrasound processing. The objective of this study is to investigate the effect of high power ultrasound and pasteurisation on the aroma profile and sensory properties of apple juice and nectar. Samples were treated according to the experimental design, with high power sonicator at ultrasound frequency of 20 kHz under various conditions (treatment time: 3, 6 and 9 min, sample temperature: 20, 40 and 60 °C, and amplitude: 60, 90 and 120 μm). The aromatic profiles of juices showed that, compared to the untreated samples of juices and nectars, ultrasonic treatment led to the formation of new compounds (which were not present in the untreated samples) or to the disappearance of compounds that were found in the untreated samples. Samples treated at the highest amplitude (120 μm) were used for evaluation and comparison with untreated and pasteurised samples using electronic tongue study. Principal component analysis confirmed the results of electronic tongue study, which showed that the ultrasound-treated and pasteurised juices had different scores compared to the untreated samples.

Key words: high power ultrasound, apple juice and nectar, aroma profile, sensory properties, electronic tongue

Introduction

Ultrasonication is a nonthermal method of food processing that has the advantage of preserving fruit juices without causing the common side effects associated with conventional heat treatments (1–5). Applications of ultrasound in the processing and the effects of sonication on fruit juices have been studied (6–8). Sonication is a simple and effective method, which is favourable in relation to pasteurisation, because the juices retain their original char-

acteristics (9,10). Sonication technology can improve the food production through reduced processing time, higher throughput and lower energy consumption (5,11). 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 mainly been attributed to physical (cavitation, mechanical effects or micromechanical shocks) and/or chemical changes due to formation of free radi-

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cals (H^+ and OH^- due to sonochemical reaction) formed by the decomposition of water inside the oscillating bubbles. Many researchers have demonstrated reduced detrimental effects on the quality or nutritional parameters including ascorbic acid content in orange juice (8, 12,13), ascorbic acid and anthocyanin content in strawberry (7) and blackberry juices (9). This positive effect of ultrasound is assumed to be due to the effective removal of occluded oxygen from the juice (6).

Today, consumers make demands on the quality, flavour and taste of different kinds of fruit juices (14). Therefore, soft drink manufacturers are trying to keep very high quality standards of their products including the taste. To make sure that juices and nectars meet required chemical and physical parameters, the beverage industry uses sophisticated equipment and technical expertise in sample preparation and analysis (15). Apart from that, for sensory analysis, industry needs trained technicians – panelists, including a substantial amount of resources, time and cost (16,17). Thus, it is important to quickly develop and test new methodologies that will ensure reliable and low-cost alternative to these costly and lengthy procedures. Electronic tongues of several types (potentiometric, voltammetric and impedance) may represent such alternatives (17,18). Amongst a number of applications (19,20), the instrument has already been used to test several juices with a combination of a gas sensor array and voltammetric electronic tongue (20). An electronic tongue functions by combining signals from non-specific and overlapping sensors with pattern recognition methods. The interfacing and conditioning circuits are handled by computer software.

The purpose of this investigation is to examine the influence of high power ultrasound and pasteurisation on the changes in the aroma profile and sensory attributes of apple juice and nectar.

Materials and Methods

Apple juice and nectar preparation

Based on the national regulation for the production of fruit juices and complementary products (21), two different apple juices were made. Pure (100 %) apple juice and 50 % apple nectar were made with minimum of 11.2 °Bx. The composition of pure (100 %) apple juice (in mg/L) was: concentrated fruit juice 168, sugar 0, citric acid 0, water 881, and that of 50 % apple nectar: concentrated fruit juice 84, sugar 59, citric acid 3, and water 927. Untreated juice and nectar samples were denoted A1.0 and A2.0, respectively, pasteurised samples A1.P and A2.P, respectively, and the ultrasound-treated ones as A1.1–A1.16 and A2.1–A2.16, respectively (Table 1).

Experimental methods

In this study, the experiment was designed in STATGRAPHICS Centurion (StatPoint Technologies, Inc., Warrenton, VA, USA) software. The experiment consisted of 16 experimental trials (Table 1). The independent variables were amplitude: X_1 (μm), temperature: X_2 ($^\circ\text{C}$) 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). The replication of the central point was made to get good estimate of the experimental error. Repetition experiments were carried out in the order of runs designed by the program. The designs were based on the face-centred central composite design with two centre points (22,23). The total number of experiments of the designs (N) can be calculated as follows:

$$N = N_i + N_o + N_j \quad /1/$$

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

Design matrix for the experiment and the regression model proposed for the response is given below (24):

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

where β_0 is the value of the fixed response at the central point of the experiment, which is the point (0, 0, 0); and β_i , β_{ii} and β_{ij} are the linear, quadratic and cross-product coefficients, respectively. In order to demonstrate the significant effects, three-dimensional fitted surfaces were drawn (25). 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 in the aromatic profiles of juices and nectars ($p < 0.05$) after the applied treatments (pasteurisation or ultrasound) and in the values of specific compounds after treatments, compared to the untreated samples.

High power ultrasound treatments

Apple 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, CT, USA), set at 600 W, 20 kHz and 12–260 μm with a 12.7-mm diameter probe, was introduced into the vessel. Ultrasonication was carried out at an amplitude of 60, 90 and 120 μm . Juice and nectar samples were treated by ultrasounds for 3, 6 and 9 min at 20, 40 and 60 $^\circ\text{C}$. Temperature of the sample before (T_1) and after (T_2) ultrasound treatment is given in Table 1. Energy consumption (E/J) by ultrasound device was read after each ultrasound treatment and is also given in Table 1. Overheating of the samples (temperature controlled by thermocouple ± 3 $^\circ\text{C}$) during the ultrasound treatment was prevented by ice-water cooling of the treatment chamber. For this study, 16 samples of juices and 16 samples of nectars were ultrasonically treated (Table 1).

Determination of acoustic power, intensity and density

The most commonly applied method for determining the power from an acoustic horn absorbed into an aqueous solution is the calorimetric technique described by Margulis and Margulis (26). This method involves applying ultrasound (for approx. 3 min) to a known volume of water while monitoring the change in temperature with time at various ultrasonic amplitudes. The ultrasonic power can be determined from the following equation:

$$P = m \times c_p \times \left(\frac{\partial T}{\partial t} \right)_{t=0} \quad /3/$$

Acoustic intensity was calculated using the following equation:

$$AI = P/A \quad /4/$$

Acoustic density was calculated according to the following equation:

$$\delta = P/V \quad /5/$$

where P is the ultrasonic power (W), m is the mass of the sample (kg), c_p is the specific heat capacity of apple juices (3.368 kJ/(kg·°C)), ∂T is the difference in temperature between the liquid and environment (°C), t is time (min), AI is the ultrasonic intensity (W/cm²), A is the surface area of the probe (cm²), δ is acoustic density (W/cm³) and V the volume of the sample (cm³).

Pasteurisation procedure

For comparison of the achieved effects of ultrasound on the investigated parameters, parallel pasteurisation

Table 1. Experimental design of pasteurisation and ultrasound treatment (HPU) of apple juices and nectars

Treatment	Amplitude μm	T °C	t min	Sample	T ₁ °C	T ₂ °C	P W	E J	AI W/cm ²	δ W/cm ³
–	–	–	–	A1.0	–	–	–	–	–	–
Pasteurisation	–	80	2	A1.P	80	81	–	–	–	–
HPU	90	60	6	A1.1	61	60	56	20.13	48.15	0.61
HPU	60	60	9	A1.2	59	60	42	23.28	46.58	0.59
HPU	90	40	3	A1.3	41	40	62	11.26	32.37	0.41
HPU	90	40	6	A1.4	40	39	62	22.32	31.58	0.40
HPU	120	40	6	A1.5	41	40	79	28.66	32.37	0.41
HPU	120	20	3	A1.6	21	24	65	14.75	16.58	0.21
HPU	120	60	3	A1.7	59	58	74	12.84	46.58	0.59
HPU	90	40	6	A1.8	40	39	61	22.07	31.58	0.40
HPU	60	60	3	A1.9	61	58	43	7.62	48.15	0.61
HPU	60	40	6	A1.10	39	40	46	16.92	30.79	0.39
HPU	120	20	9	A1.11	21	23	67	24.34	16.58	0.21
HPU	60	20	3	A1.12	21	24	50	8.95	16.58	0.21
HPU	90	20	6	A1.13	21	18	62	23.25	16.58	0.21
HPU	120	60	9	A1.14	60	59	70	38.99	47.36	0.60
HPU	90	40	9	A1.15	39	39	61	33.27	30.79	0.39
HPU	60	20	9	A1.16	21	24	63	17.05	16.58	0.21
–	–	–	–	A2.0	–	–	–	–	–	–
Pasteurisation	–	80	2	A2.P	80	81	–	–	–	–
HPU	90	60	6	A2.1	59	60	56	20.14	46.58	0.59
HPU	60	60	9	A2.2	60	59	42	23.44	47.36	0.60
HPU	90	40	3	A2.3	40	41	60	10.82	31.58	0.40
HPU	90	40	6	A2.4	40	41	59	21.34	31.58	0.40
HPU	120	40	6	A2.5	41	39	76	27.78	32.37	0.41
HPU	120	20	3	A2.6	22	19	72	14.64	17.37	0.22
HPU	120	60	3	A2.7	59	60	71	12.77	46.58	0.59
HPU	90	40	6	A2.8	41	42	62	21.86	32.37	0.41
HPU	60	60	3	A2.9	61	59	44	7.78	48.15	0.61
HPU	60	40	6	A2.10	40	41	47	16.81	31.58	0.40
HPU	120	20	9	A2.11	21	23	70	14.40	16.58	0.21
HPU	60	20	3	A2.12	21	23	52	8.59	16.58	0.21
HPU	90	20	6	A2.13	21	23	61	22.86	16.58	0.21
HPU	120	60	9	A2.14	59	60	73	39.72	46.58	0.59
HPU	90	40	9	A2.15	40	41	62	33.44	31.58	0.40
HPU	60	20	9	A2.16	21	24	48	25.82	16.58	0.21

A1.0=untreated apple juice (100 %), A2.0=untreated apple nectar (50 %), A1.P=pasteurised apple juice sample, A2.P=pasteurised apple nectar, A1.1–A1.16=ultrasonically treated apple juice samples, A2.1–A2.16=ultrasonically treated apple nectar samples, T₁=temperature before the treatment, T₂=temperature after the treatment, P=power of the ultrasound treatment, E=energy of the ultrasound treatment, AI=acoustic intensity, δ=acoustic density

process was carried out. Pasteurisation of the samples was carried out on the heater with a magnetic mixer (IKA RTC Basic, Ika-Werke GmbH & Co. KG, Janke & Kunkel, Staufen, Germany) where the samples (100 mL) were placed in glass containers and covered with aluminium foil over hot water bath at a temperature of 80 °C for 2 min (Table 1). Pasteurised samples of apple juice were denoted A1.P and of apple nectar A2.P.

Solid phase microextraction and gas chromatography-mass spectrometry analysis of apple juices and nectars

Analyses of the extracted volatile compounds of the above samples were carried out using solid phase microextraction (SPME) fibre, and their qualification and quantification were done using gas chromatography-mass spectrometry (GC-MS). Before and after the ultrasound or pasteurisation treatments, the juice and nectar samples were homogenised, and 10 mL of each sample were placed into a 20-mL vial tightly capped with a PTFE septum. A small magnetic stirrer was placed into the homogenates during the extraction. SPME fibre coated with 100 µm of polydimethylsiloxane (PDMS; Supelco, Bellefonte, PA, USA) was preconditioned for 1 h at 250 °C before the first use. Before each extraction, the fibre was 'cleaned' (conditioned) in the GC injector for 15 min at 250 °C, and then placed above the sample. Samples were placed in a water bath at 40 °C and extracted for 30 min with stirring (27,28). After the extraction, the SPME fibre was immediately injected to 6890N gas chromatograph coupled to a 5975i mass selective detector (Agilent Technologies, Santa Clara, CA, USA). Capillary column DB-5ms, 30 m×0.25 mm, film thickness of 0.25 µm (Agilent Technologies) was used with helium as a carrier gas at a flow rate of 1.0 mL/min. The temperature of the injector, used in the splitless mode, was 220 °C and the desorption time was 4 min. Temperature programme was set at 40 °C, isothermal for 1 min, then raising to 250 °C at a rate of 5 °C/min. Final temperature was held for 4 min. The transfer line temperature was maintained at 280 °C. The mass spectra were obtained at 70 eV with a rate of 1 scan/s over the *m/z* range of 40–550. Each sample was analysed in three replicates. In-house prepared mixture of C8-C20 *n*-alkanes was run under the same chromatographic conditions to calculate the retention indices (*RI*) of the detected compounds. AMDIS 3.2 v. 2.62 programme was used for the identification of the components using NIST 2005 v. 2.0 spectral library (NIST, Gaithersburg, MD, USA) as well as for the comparison of the obtained retention indices with literature values (29 and in-house library).

The potentiometric electronic tongue

The potentiometric electronic tongue used in this study was α -ASTREE, Alpha M.O.S. Co., Toulouse, France. It included the automatic sampling system, the sensor array with the reference electrode, the signal processing unit and a personal computer with the ASTREE v. 3.0.1. software installed. It is comprised of seven potentiometric chemical sensors for food application (ZZ3401, BA3401, BB3401, CA3401, GA3401, HA3401, JB3401) based on ion sensitive field effect transistor (ISFET). Due to the poly-

mer membrane coating, sensors display sensitivity to organic acids, salts, mono- and disaccharides. Furthermore, sensors have cross-sensitivity for taste chemicals which are typically found in foodstuffs and beverages. The sensor array consisted of seven sensors coated with lipid/polymer material and a reference Ag/AgCl electrode. Sensors were conditioned with milk samples before the actual measurements were performed (19).

Measurement procedure: selected samples of apple juices and nectars were analysed: untreated samples (A1.0 and A2.0), pasteurised (A1.P and A2.P) and the ultrasound-treated samples at 120 µm amplitude, *i.e.* the highest power (A1.5, A1.11, A1.14, A2.5, A2.11 and A2.14). Three measurements were performed for each sample. The obtained data were evaluated and each day one measurement was selected according to the lowest available sensor drift value. The selected results were further analysed by principal component analysis (PCA). The acquisition time was 200 s for each sample, which was experimentally determined to be optimal for flat responses of the sensors. Between each measurement, the sensors were cleaned with deionized water. The sensor signals were acquired using the ASTREE software v. 3.0.1., installed on the PC.

Statistical analysis

The acquired data were evaluated using PCA with the embedded ASTREE software v. 3.0.1. This statistical method identifies patterns in data, and it expresses the data in such a way that it highlights their differences and similarities. It reduces the amount of data to a smaller number of derived variables which adequately represent the original data (30).

Analysis of the sensory properties of apple juices and nectars

Sensory properties of apple juices and nectars were determined by ten panelists. They evaluated the following sensory characteristics of the samples according to the hedonistic scale: taste, odour, aroma and colour (31). For odour and aroma, it was possible to give maximum 6 points, and for colour and taste maximum of 4 points, which means that the maximum possible score was 20 points. Results are expressed graphically as the mean values of all ratings for each component and the overall score.

Results and Discussion

The highest acoustic intensity (*AI*) of the ultrasound treatment (maximum power per unit area of embedded probes) was determined in samples A1.1, A1.9 and A2.9 (48.15 W/cm²), and the lowest in treatments A1.6, A1.11, A1.12, A1.16, A2.11, A2.12, A2.13 and A2.16 (16.58 W/cm²) (Table 1). The results of energy consumption during ultrasound processing of apple juice and nectar were highest in the sample A2.14 (39.724 J), and the lowest in the sample A2.9 (7.789 J) (data not shown).

Aroma profile analysis

Tables 2 and 3 show the aromatic profiles of apple juices and nectars, and the fraction of individual com-

Table 2. Aromatic profile of apple juice before treatment (A1.0), after pasteurisation (A1.P) and after ultrasonic treatments (A1.1–A1.16)

RT/min	Compound	RI	Compound in total area/%																	
			A1.0	A1.1	A1.2	A1.3	A1.4	A1.5	A1.6	A1.7	A1.8	A1.9	A1.10	A1.11	A1.12	A1.13	A1.14	A1.15	A1.16	A1.P
4.72	butyl acetate	820	n.d.	4.84	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5.04	n.d.	n.d.	n.d.	3.59	n.d.	n.d.	n.d.	n.d.	n.d.
5.52	ethyl 2-methylbutyrate	855	n.d.	14.50	n.d.	1.72	1.36	1.42	1.29	6.28	16.90	n.d.	2.80	0.78	14.13	5.91	0.49	1.18	2.38	11.98
5.61	ethyl 3-methylbutyrate	859	n.d.	9.23	n.d.	n.d.	n.d.	0.98	n.d.	3.92	9.66	n.d.	1.57	n.d.	7.61	3.81	n.d.	n.d.	n.d.	8.34
5.99	1-hexanol	874	n.d.	6.55	n.d.	0.86	n.d.	0.96	n.d.	3.08	0.02	n.d.	1.04	n.d.	3.49	n.d.	n.d.	n.d.	n.d.	9.27
6.19	3-methyl-1-butyl acetate	881	n.d.	3.77	n.d.	n.d.	n.d.	0.70	n.d.	2.33	4.18	n.d.	1.08	3.56						
8.95	isohexyl acetate	980	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.85	n.d.									
9.59	hexyl acetate	999	n.d.	0.66	n.d.	0.70	0.54	0.58	0.57	1.01	0.77	n.d.	0.58	n.d.	0.77	1.00	n.d.	0.55	0.79	0.57
9.77	<i>cis</i> -hexane-3-en-1-yl acetate	1006	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.39	n.d.								
10.00	ethyl hexanoate	1015	17.43	37.86 ^b	17.43 ^a	39.44 ^b	31.22 ^b	33.47 ^b	33.31 ^b	n.d.	42.76 ^b	12.43 ^b	36.41 ^b	26.71 ^b	43.08 ^b	55.01 ^b	14.00 ^b	34.40 ^b	47.82 ^b	35.17 ^b
10.93	ethyl hexan-2-enoate	1047	1.07	2.16	1.07	2.38	1.84	2.01	1.89	3.43	2.34	0.79	2.01	1.79	2.23	2.68	0.02	2.13	2.79	2.48
12.39	2,3-dimethyl-2-hexanol	1093	0.44	n.d.	0.44	n.d.														
12.61	undecane	1100	0.62	0.81	0.62	1.08	0.74	1.16	1.00	1.09	0.89	0.45	1.18	1.04	1.05	1.30	n.d.	1.18	1.79	0.53
12.73	nonanal	1105	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.19
14.69	5-decanone	1173	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.21	n.d.
15.26	butyl hexanoate	1191	1.12	0.70	1.12	1.59	1.12	1.28	1.40	n.d.	0.77	0.91	1.24	1.12	0.97	1.42	0.52	1.38	1.91	0.60
15.69	decanal	1205	n.d.	0.38	n.d.	0.31	n.d.	0.37	n.d.	0.38	0.28	n.d.	n.d.	0.32	n.d.	n.d.	0.51	n.d.	n.d.	0.60
16.51	2-methylbutyl hexanoate	1236	2.49	1.19	2.49	2.74	1.92	n.d.	2.54	2.60	1.27	1.83	2.31	2.00	1.59	2.41	0.99	2.48	3.36	1.38
16.68	3-methylbutyl hexanoate	1242	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.55	1.71	n.d.	n.d.	1.29	n.d.	0.89	n.d.	0.69	n.d.	n.d.	0.74
17.54	1-decanol	1273	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.16	0.24	n.d.	n.d.	n.d.	n.d.	n.d.	0.23	0.31	n.d.
18.00	hexyl 3-methylbutyrate	1288	n.d.	n.d.	n.d.	n.d.	n.d.	0.29	n.d.	0.45	n.d.									
20.20	2-butyl 2-octenal	1371	18.25	7.27 ^b	18.25 ^a	20.66 ^b	15.64 ^b	16.99 ^b	19.32 ^a	18.35 ^a	7.16 ^b	17.62 ^a	17.10 ^b	16.16 ^b	9.59 ^b	16.51 ^b	8.52 ^b	18.91 ^a	25.09 ^b	7.32 ^b
20.41	dodecanone	1379	3.83	1.39	3.83	3.21	2.63	3.26	2.70	4.01	1.15	3.10	2.32	3.43	1.07	2.21	2.37	3.24	3.16	2.49
20.58	hexyl hexanoate	1385	2.78	0.83	2.78	2.47	1.84	2.14	2.30	2.02	0.82	1.89	2.12	2.07	1.16	1.97	1.13	2.27	3.19	0.88
22.17	2-hexenyl 2-hexenoate	1448	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.69	n.d.										
22.84	1-dodecanol	1474	n.d.	0.70	n.d.	0.56	0.44	n.d.	n.d.	0.60	0.28	0.53	n.d.	n.d.	0.28	n.d.	0.81	n.d.	n.d.	1.22
26.45	ethyl dodecanoate	1625	41.38	n.d.	41.38 ^a	17.99 ^b	37.24 ^b	32.48 ^b	27.45 ^b	40.40 ^a	3.75 ^b	54.78 ^b	25.50 ^b	40.28 ^a	7.41 ^b	6.28 ^b	61.85 ^b	23.39 ^b	n.d.	7.98 ^b
27.28	tetradecanol	1662	3.78	3.26	3.78	2.79	1.75	1.81	1.82	3.20	0.96	3.01	1.74	2.51	0.73	1.68	2.79	2.64	3.24	0.49
28.26	2-ethylhexyl benzoate	1705	n.d.	n.d.	n.d.	0.48	n.d.	0.43	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.						
29.05	isoamyl cinamate	1742	n.d.	0.44	n.d.	0.30	0.31	n.d.	0.29	0.64	0.22	0.48	0.31	0.28	n.d.	0.32	n.d.	0.40	0.65	0.73
30.28	2-ethylhexyl salicylate	1800	5.20	0.92	5.20	n.d.	0.71	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.							
30.75	ethyl myristate	1823	n.d.	1.12	n.d.	0.33	0.38	n.d.	1.14	1.33	0.24	n.d.	n.d.	n.d.	0.48	0.98	1.31	0.15	1.32	
30.82	isohexadecanol	1826	1.02	n.d.	1.02	0.41	0.33	n.d.	0.44	1.45	n.d.	0.85	0.48	n.d.	0.38	n.d.	1.15	1.29	1.14	1.62
31.09	diterpene	1840	n.d.	n.d.	n.d.	n.d.	0.41	0.39	n.d.											
Total count/%			99.41	98.58	99.41	100.00	99.72	100.00	100.00	98.70	100.00	98.88	100.00	99.63	100.00	99.93	96.82	96.97	99.53	99.46
esters			66.27	76.18	66.27	69.80	77.40	75.06	73.29	65.21	88.87	73.09	76.14	75.47	83.42	80.81	79.70	68.17	63.98	74.41
alcohols			4.80	3.96	4.80	3.76	2.52	1.81	2.26	5.26	1.40	4.62	2.22	2.51	1.39	1.68	4.75	4.16	4.69	3.33
aldehyds			18.25	7.64	18.25	20.96	15.64	17.36	19.32	18.73	7.44	17.62	17.10	16.48	9.59	16.51	9.03	18.91	25.09	8.11
ketones			3.83	1.39	3.83	3.21	2.63	3.26	2.70	4.01	1.15	3.10	2.32	3.43	1.07	2.21	2.37	3.24	3.37	2.49
acids			0.00	1.12	0.00	0.33	0.38	0.00	1.14	1.33	0.24	0.00	0.00	0.00	0.00	0.48	0.98	1.31	0.15	1.32
others			6.26	8.29	6.26	1.94	1.14	2.51	1.30	4.17	0.91	0.45	2.23	1.75	4.54	1.25	0.00	1.18	2.23	9.80

^anot statistically significant difference, ^bstatistically significant difference ($\alpha=0.05$), RT=retention time, RI=retention index, n.d.=not determined

Table 3. Aromatic profile of apple nectar before treatment (A2.0), after pasteurisation (A2.P) and after ultrasonic treatments (A2.1–A2.16)

RT/min	Compound	RI	Compound in total area/%																		
			A2.0	A2.1	A2.2	A2.3	A2.4	A2.5	A2.6	A2.7	A2.8	A2.9	A2.10	A2.11	A2.12	A2.13	A2.14	A2.15	A2.16	A2.P	
4.72	butyl acetate	820	n.d.	n.d.	n.d.	n.d.	n.d.	2.91	3.79	n.d.	3.13										
5.52	ethyl 2-methylbutyrate	855	3.04	1.59	0.63	n.d.	0.60	9.73	13.99	2.20	1.36	0.72	2.27	1.30	1.12	0.73	n.d.	1.07	1.14	9.97	
5.61	ethyl 3-methylbutyrate	859	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7.08	n.d.	4.95										
5.99	1-hexanol	874	n.d.	n.d.	n.d.	n.d.	n.d.	4.59	4.40	0.95	n.d.	n.d.	0.74	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	3.90	
6.19	3-methyl-1-butyl acetate	881	n.d.	n.d.	n.d.	n.d.	n.d.	2.71	3.36	n.d.	n.d.	n.d.	0.74	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.42	
9.59	hexyl acetate	999	0.59	n.d.	n.d.	n.d.	n.d.	0.49	0.63	0.34	0.52	n.d.	0.37	0.42							
9.77	<i>cis</i> -hexan-3-en-1-ol acetate	1006	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.30	n.d.	0.23										
9.99	ethyl hexanoate	1015	47.79	29.94 ^b	13.64 ^b	23.21 ^b	20.87 ^b	31.45 ^b	38.59 ^b	18.74 ^b	32.31 ^b	19.12 ^b	29.46 ^b	32.95 ^b	36.73 ^b	27.78 ^b	27.26 ^b	30.09 ^b	25.76 ^b	25.23 ^b	
10.93	ethyl hexan-2-enoate	1047	2.23	1.56	0.73	1.30	1.22	1.67	1.93	0.99	1.69	1.05	1.55	1.95	1.96	1.50	1.45	1.77	25.81	1.30	
12.07	unknown ester	1084	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.82	n.d.	0.80	n.d.									
12.39	2,3-dimethyl-2-hexanol	1093	n.d.	n.d.	0.17	0.39	0.39	n.d.	0.51	n.d.	0.61	n.d.									
12.61	undecane	1100	0.75	0.43	0.23	0.26	0.31	0.46	0.45	0.15	0.52	0.26	0.44	n.d.	n.d.	0.97	n.d.	0.53	0.35	0.25	
15.26	butyl hexanoate	1191	1.76	1.08	0.50	1.00	0.87	0.54	0.71	0.46	1.12	0.79	0.93	n.d.	1.67	1.46	n.d.	1.20	0.92	0.46	
15.69	decanal	1205	n.d.	0.48	n.d.	0.55	0.40	0.40	n.d.	0.21	0.37	0.38	n.d.	n.d.	0.63	0.80	1.22	n.d.	0.46	0.16	
16.51	2-methylbutyl hexanoate	1236	3.11	1.83	0.96	1.82	1.61	0.88	1.18	0.83	1.93	1.51	1.59	2.55	2.99	2.59	2.54	2.11	1.54	0.95	
16.68	3-methylbutyl hexanoate	1242	1.78	n.d.	n.d.	1.21	n.d.	0.54	n.d.	0.52	1.25	n.d.	1.00	1.57	1.90	1.79	n.d.	1.33	n.d.	n.d.	
17.54	1-decanol	1273	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.09	0.22	n.d.									
20.19	2-butyl 2-octenal	1371	21.93	12.00 ^b	6.16 ^b	15.54 ^b	12.75 ^b	4.98 ^b	7.56 ^b	5.34 ^b	15.15 ^b	10.71 ^b	11.94 ^b	21.10 ^a	26.02 ^b	22.18 ^a	16.97 ^b	16.84 ^b	12.90 ^b	5.25 ^b	
20.41	dodecanone	1379	2.34	2.63	1.31	2.83	2.35	1.11	0.98	1.20	2.51	1.90	1.75	3.02	3.32	2.80	4.36	3.02	2.07	1.13	
20.58	hexyl hexanoate	1385	2.40	1.78	0.88	1.92	1.69	0.79	0.95	0.72	1.98	1.30	1.56	2.79	3.37	2.98	2.79	2.31	1.67	0.72	
21.20	dodecanol	1408	n.d.	n.d.	0.18	0.40	0.35	0.23	n.d.	n.d.	n.d.	0.25	n.d.	0.18							
21.77	2-dodecanol	1432	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.18	
21.95	isopropyl decanoate	1439	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.66	n.d.	
22.15	2-hexenyl 2-hexenoate	1448	n.d.	n.d.	n.d.	0.99	1.09	0.34	n.d.	0.48	n.d.	0.61	n.d.	n.d.	2.06	1.58	2.08	n.d.	1.06	0.32	
22.48	unknown ester	1460	n.d.	n.d.	n.d.	n.d.	n.d.	0.30	0.22	0.45	0.49	0.33	n.d.	0.71	1.03	0.57	1.00	n.d.	0.44	n.d.	
22.83	1-dodecanol	1474	1.17	0.69	0.50	0.90	0.89	0.72	0.54	0.46	1.38	0.68	n.d.	n.d.	n.d.	1.68	1.31	0.81	0.81	0.41	
23.50	butyl hydroxytoluen	1500	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.27	n.d.	n.d.	n.d.	n.d.	n.d.	0.49	n.d.	n.d.	n.d.	n.d.	
25.78	unknown ester	1596	n.d.	n.d.	0.27	n.d.															
26.44	ethyl dodecanoate	1625	0.11	n.d.	66.49 ^b	33.20 ^b	37.52 ^b	25.65 ^b	8.40 ^b	54.69 ^b	20.63 ^b	43.44 ^b	35.79 ^b	20.41 ^b	n.d.	17.97 ^b	n.d.	21.89 ^b	16.45 ^b	34.00 ^b	
27.28	tetradecanol	1662	3.67	37.38	4.18	5.56	6.09	3.49	1.76	5.10	6.05	6.93	6.15	5.06	10.66	5.68	20.97	7.18	4.01	1.69	
27.59	<i>n</i> -hexyl salicylate	1676	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.35	n.d.										
28.26	2-ethylhexyl benzoate	1705	n.d.	n.d.	0.39	n.d.	0.86	0.41	n.d.	n.d.	n.d.	n.d.	0.54	n.d.	n.d.	n.d.	1.75	n.d.	n.d.	n.d.	
29.05	isoamyl cinamate	1742	0.71	0.64	0.52	0.89	0.84	0.39	n.d.	0.52	0.90	0.62	n.d.	0.51	1.15	0.69	2.03	1.24	0.54	0.31	
30.28	2-ethylhexyl salicylate	1800	n.d.	n.d.	n.d.	n.d.	3.36	0.79	n.d.	n.d.	1.13	n.d.	n.d.	1.24	n.d.	1.47	1.51	n.d.	n.d.	0.47	
30.75	tetradecanoic acid	1823	2.19	5.39	0.71	1.15	1.57	0.53	0.39	1.79	1.80	2.02	1.89	1.61	0.79	1.65	4.68	2.21	0.57	0.28	
30.83	ethyl myristate	1826	0.93	n.d.	0.29	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.39	n.d.								
31.08	diterpene	1840	1.87	0.88	0.85	2.04	1.39	0.45	n.d.	0.64	1.17	1.53	n.d.	0.72	1.11	0.90	4.37	1.76	n.d.	0.96	
32.79	ethyl pentanoate	1924	n.d.	0.46	n.d.																
Total count/%			98.37	98.76	99.59	95.16	97.02	96.55	98.54	97.49	95.89	94.54	98.34	97.49	96.51	96.58	96.66	95.86	98.53	99.27	
esters			67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61	67.61
alcohols			5.77	38.06	5.32	7.77	7.71	9.02	7.22	6.60	8.26	8.27	6.89	5.06	10.66	5.68	22.65	8.49	4.82	6.36	
aldehyds			21.93	12.48	6.16	16.09	13.15	5.38	7.56	5.55	15.52	11.09	11.94	21.10	26.65	22.98	18.18	16.84	13.36	5.41	
ketones			2.34	2.63	1.31	3.82	3.43	1.75	1.40	2.13	3.00	2.84	1.75	3.73	6.41	4.95	7.45	3.02	3.57	1.64	
acids			2.19	5.39	0.71	1.15	1.57	0.53	0.39	1.79	1.80	2.02	1.89	1.61	0.79	1.65	4.68	2.21	0.57	0.28	
others			4.25	1.97	1.08	6.61	3.55	2.72	1.00	3.39	2.67	6.75	0.44	0.72	1.11	3.37	5.76	2.29	0.35	1.42	

^anot statistically significant difference, ^bstatistically significant difference ($\alpha=0.05$), RT=retention time, RI=retention index, n.d.=not determined

pounds in untreated, pasteurised and ultrasonically treated samples. From both tables it is evident that, in comparison with the untreated samples of apple juice and nectar, ultrasonic treatment leads to the formation of new compounds (which are not present in the untreated sample) or the disappearance of compounds that are present in the untreated samples. It was found that pasteurisation leads to the development of more aromatic compounds in the samples of apple juice and nectar in comparison with the untreated samples. Changes in the composition of aromatic compounds derive from the extreme physical conditions that occur inside the cavitation bubbles after the collapse at the micro-level (32) as a result of several sonochemical reactions that occur simultaneously or separately. Cavitation generates high local temperature, pressure and mechanical action between the solid and liquid media, which causes the chemical changes in the samples (33).

For example, in apple juice the ester ethyl dodecanoate has the largest fraction in the untreated sample (41.38 %). In pasteurised samples, the fraction of this compound is 7.98 % ($p < 0.05$), and after the ultrasound treatment at the amplitude of 120 μm at 60 °C for 9 min (sample A1.14) its fraction increased to 61.85 % ($p < 0.05$), while the smallest fraction (3.75 %; $p < 0.05$) was found after the ultrasonic treatment at 90 μm and 40 °C for 6 min (sample A1.8). In the untreated apple nectar, ethyl hexanoate had the largest fraction (47.79 %), which decreased ($p < 0.05$) after ultrasound treatment and pasteurisation (25.23 %), with the greatest reduction in it after the ultrasonic treatment at 120 μm and 60 °C for 3 min (sample A2.7). On the other hand, it was found that the fraction of ethyl dodecanoate, which is a minor compound in the untreated apple nectar sample (0.11 %), after ultrasonic treatment of sample A2.2 (60 μm /60 °C/9 min) increased to 66.49 % ($p < 0.05$).

The reason for these results is in the combination of the properties of acoustic waves and the chemical changes caused by imploding cavitation bubbles and subsequent hydrolysis of water to form free radicals, which participate in chemical reactions. Ultrasound can induce rapid and complete degassing, run a variety of chemical reactions of free ions (radicals), enhance the reaction of polymerization/depolymerization, and improve the rate of expansion and many other effects (34). The degradation of aromatic compounds may be due to the sonolysis of water as a consequence of cavitation, which encourages the formation of hydroxyl radicals that leads to chemical degradation (35). Also, previous research has shown by sensorial and chemical analyses that off-flavours are generated when ultrasound is used for cutting, crystallisation or emulsifying (2). Vercet *et al.* (11) found that the formation of hydroxyl ions (OH^-) increases linearly with the increase of amplitude of the ultrasonic level and decreases with the increase of temperature. The major difference between the apple juice and the nectar samples is in the fraction of fruit concentrate (half of the amount in nectar than in juice) and the sugar, which was added to nectar only. This could also be the reason for differences in aroma profile. There is also a link between the increase of ultrasound power (higher amplitudes and higher temperatures) and the degradation of antioxidants, which prevent the oxidative degradation of aroma compounds (2).

The greater number of OH^- ions at 70 than at 130 °C indicates that the number of cavitation bubbles decreases with the increase of temperature, because of the cushioning effect of vapour inside the bubble. Cavitation thermolysis may produce hydroxyl radicals and hydrogen atoms, and may be followed by the formation of hydrogen peroxide, or in the absence of oxygen radicals, hydroperoxide radicals (36,37). The production of H_2O_2 during the sonication is also temperature dependent, decreasing with the increase of temperature. Increasing the temperature during the sonication allows reducing the cavitation threshold, although the maximum temperature and pressure at which cavitation bubbles collapse decrease (5). Several mechanisms may act simultaneously when ultrasound is applied in a liquid medium: thermal effects of the implosion of bubbles, mechanical stresses produced by microstreaming, implosion and shock waves, and the production of free radicals. However, the formation of radicals was considered as the most likely mechanism of compound degradation (11,37). With regard to the type of individual compounds that make up the aroma profile of the analysed juices, the identified components are divided into groups of esters, alcohols, aldehydes, ketones, acids and other compounds. In the untreated and pasteurised apple juice and nectar samples, it was found that the aroma profile consists mainly of esters. Aroma compounds before and after sonication or pasteurisation were influenced by three investigated factors, *i.e.* ultrasound amplitude level, sonication time and temperature.

The results of potentiometric electronic tongue analyses of apple juices and nectars

The main focus of the electronic tongue study of ultrasound-treated and pasteurised samples was to evaluate the performance of the electronic tongue in classification of different samples and to qualitatively compare differences between the untreated and treated samples. Untreated (A1.0 and A2.0), pasteurised (A1.P and A2.P) and ultrasound-treated samples at the highest amplitude (A1.5, A1.11, A1.14, A2.5, A2.11 and A2.14) were compared. The interaction of compounds in the sample and the sensitive coating of sensors generates a potential on the membranes which is measured between the sensors and the reference electrode. The potentials depend on the nature and concentration of the ionic species in a solution as well as on the medium and the type of electrodes (38).

Figs. 1 and 2 show the PCA plot of the effect of the first two discriminant functions in the classification of different samples considered. Apple juice samples A1.5 and A1.11 (Fig. 1a) are 'similar', but in the diagonally opposite quadrant from the untreated sample A1.0, so there is a difference observed by the electronic tongue. This means that ultrasound-treated samples are different compared to the untreated sample A1.0 and also to sample A1.P, which is in the opposite quadrant. Since pasteurised sample A1.P is in a different quadrant from untreated and ultrasound-treated samples there is qualitative difference among these samples. Sample A1.14 (120 μm /60 °C/9 min) is in the same quadrant and close to sample A1.0, so it can be concluded that there is least significant difference between it and the untreated sample (they are 'similar'). Fig. 1b shows the response of seven sensors

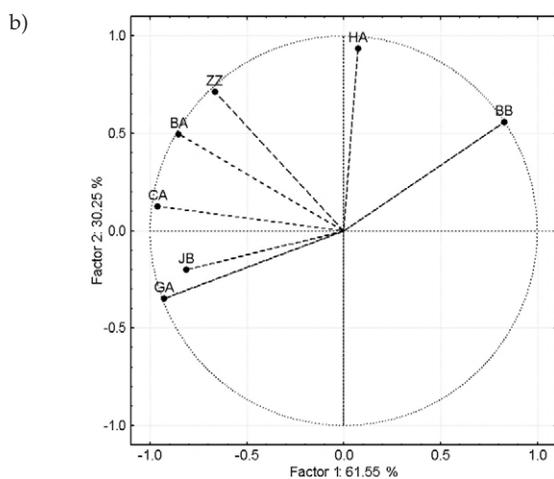
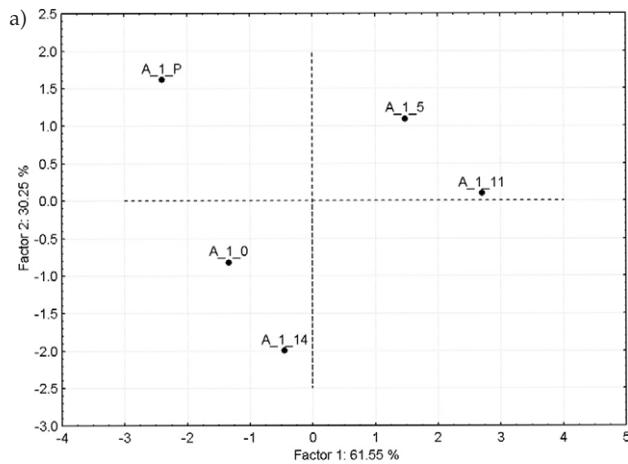


Fig. 1. Principle component analysis analysis of pure (100 %) apple juice samples: a) plot of the effect of the first two discriminant functions in the classification of different samples considered (A1.0=untreated, A1.P=pasteurized, A1.5, A1.11 and A1.14=ultrasonically treated), and b) the response of 7 sensors that were used for the analysis

that were used for the analysis. PCA enables to visualize the information detected by the sensor array in a two-dimensional space. Sensor array response in all complex liquids appeared to be reproducible and stable enough to provide adequate integral information about the analyzed media. The most stable sensor array output has been observed in soft drinks. Stability and long-term durability of sensor array output have always been sufficient enough to give valuable information about the analyzed complex liquid. Electronic sensors have high correlations with specific sensory attributes, and thus have a potential to predict them. Those that are labelled as 'similar' have a positive correlation and those that are labelled as 'opposite' have a negative correlation.

Fig. 2a shows qualitative response in differences among the untreated (A2.0), pasteurised (A2.P) and ultrasound-treated samples (A2.5, A2.11 and A2.14) of apple nectars. These samples were selected to observe the impact of the most powerful ultrasound treatment (at 120 μm amplitude). It can be observed from the figure that the untreated sample (A2.0) is in the first quadrant and A2.14 sample is (the same as for the apple juice sample)

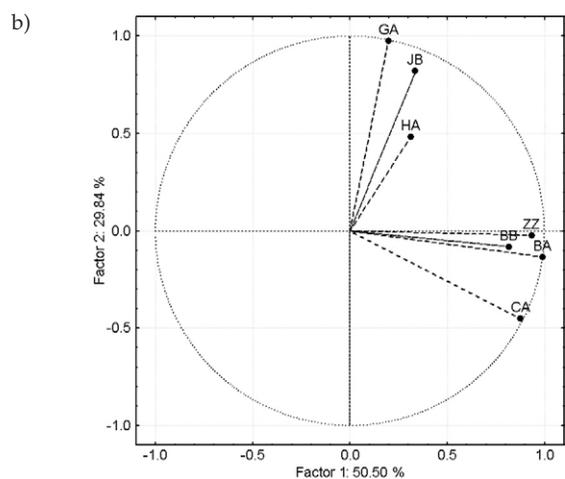
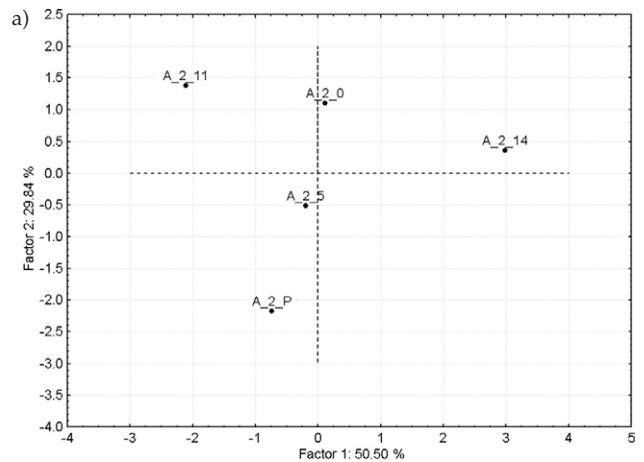


Fig. 2. Principle component analysis of apple nectar samples with 50 % fruit: a) plot of the effect of the first two discriminant functions in the classification of different samples considered (A2.0=untreated, A2.P=pasteurised, A2.5, A2.11 and A2.14=ultrasonically treated), and b) the response of 7 sensors that were used for the analysis

closest to the untreated sample, showing least significant difference (they are 'similar'). Sample A2.5 is in the same quadrant as the pasteurised sample A2.P, so there is least significant difference between them (they are 'similar'). Sample A2.11 is the only one in the second quadrant so the 'sensing' of that sample is completely opposite to other four samples. Fig. 2b shows the response of seven sensors that were used for the analysis. These are potentiometric sensors with a membrane that gives each sensor a specific sensitivity and selectivity. The final information, either quantitative or qualitative, about complex systems was obtained by data processing. Only harmonic combination of sensor array and pattern recognition tool makes the whole device perform as electronic tongue (39). The sensors measure dissolved organic compounds in liquids including taste and flavour compounds.

The results of analysis of sensory quality of apple juices and nectars

The results of the evaluation of sensory properties (taste, odour, aroma and colour) are shown in Figs. 3 and 4. It can be concluded from sensory evaluation that ultra-

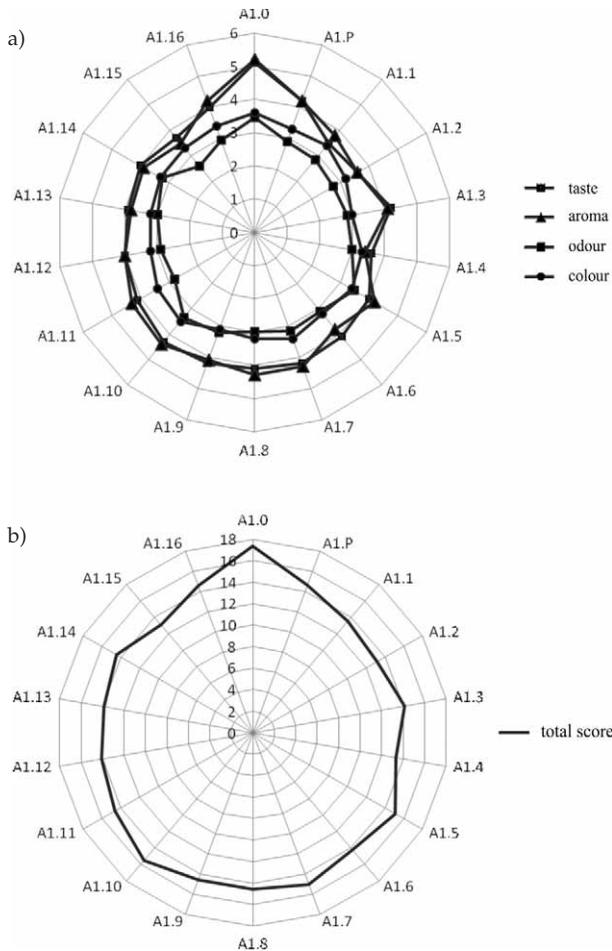


Fig. 3. Graphical plot of sensory analysis of: a) specific parameters (taste, aroma, odour, colour), and b) total score of tested apple juice samples
A1.0=untreated; A1.P=pasteurised, A1.5, A1.11, A1.14=ultrasonically treated apple juice

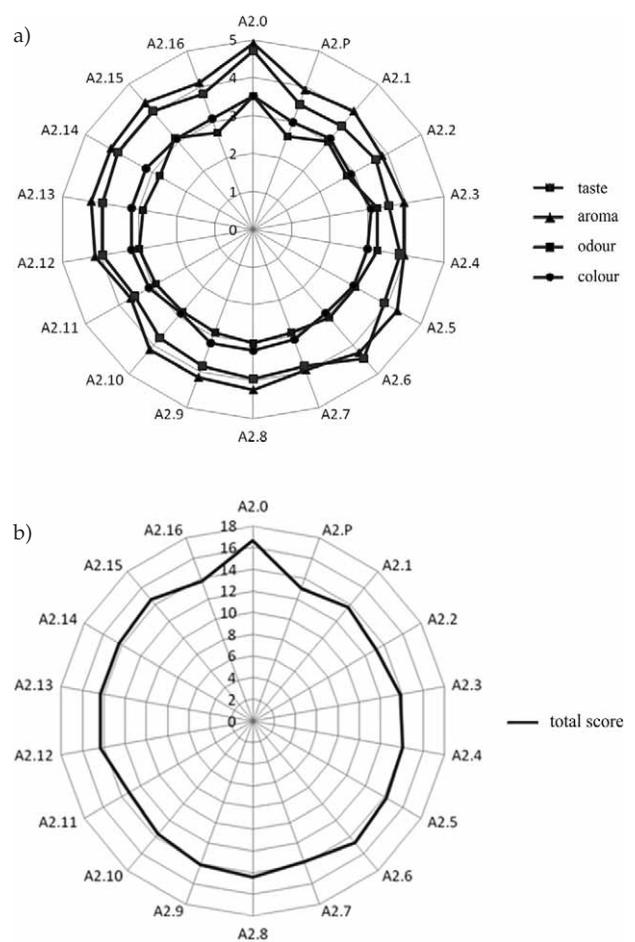


Fig. 4. Graphical plot of sensory analysis of: a) specific parameters (taste, aroma, odour, colour), and b) total score of tested apple nectar samples
A2.0=untreated, A2.P=pasteurised, A2.5, A2.11, A2.14=ultrasonically treated apple nectar

sonically treated and pasteurised juices are evaluated with different scores in comparison with the untreated samples. The differences in ratings of taste, odour, aroma and colour depend on the applied ultrasound treatment and on the type of juice. The best accepted are ultrasonically treated apple juice at 60 μm and 40 $^{\circ}\text{C}$ for 6 min (sample A1.10) and apple nectar at 120 μm and 20 $^{\circ}\text{C}$ for 3 min (sample A2.6). From the sensory point of view, the ultrasonic treatment caused a statistically significant decrease in all tested sensory parameters (colour, odour, taste, aroma and overall quality), but without sensory rejection of the product (16). Cavitation induced by ultrasound treatment has shown to contribute to the changes in colour and taste of fruit juices. The positive effect of ultrasound for preservation of similar processed products compared to raw products is attributed to the removal of oxygen (5,7,10). During cavitation, implosion and explosion of vapour bubbles and degassing of fluid take place, thus oxygen can be liberated from the media. Ultrasound treatment of fruit juices showed minimal effect on colour change during processing in relation to heat treatment (pasteurisation). It is assumed that this positive effect of ultrasound compared to heating is due to the efficient removal of dissolved oxygen from the

juice (6), because this is a critical parameter that affects oxidation. Juice colour is mainly influenced by the presence of natural pigments, depending on the degree of fruit maturity, storage conditions, enzyme activity and microbial contamination (40). Possible changes in the odour and taste of fruit juices treated with ultrasound can be attributed to the rapid isomerization of compounds and oxidation (which occur as a result of interaction with free radicals) generated during the treatment. Caminiti *et al.* (41) found that a combination of ultraviolet light and manothermosonication (MTS) that was applied to apple and cranberry juices had a negative effect on the odour and taste of the product. Gómez-López *et al.* (42) also found that a continuous ultrasound processing at the amplitude of 89.25 μm , for a maximum of 10 min affected colour, aroma and flavour of orange juice. Walkling-Ribeiro *et al.* (13) described the effect of combined thermosonication (55 $^{\circ}\text{C}/10$ min) and processing with pulsed electric field (40 kV/cm, 100 ms) on the sensory properties of orange juice, and concluded that there were no significant differences in any properties in respect to the heat-treated (94 $^{\circ}\text{C}/26$ s) orange juice. Panelists who performed sensory evaluation of untreated, pasteurised and ultrasonically treated apple juices

and nectars did not find metallic taste in ultrasonically treated samples as compared to other fruit juices (data not shown). However, they determined differences among untreated, pasteurised and ultrasound-treated apple juices and nectars. Vercet *et al.* (11), who studied MTS of apple juice and cranberry, suggest that metallic taste could appear due to changes caused by free radicals produced during MTS.

Conclusions

The purpose of this investigation was to examine the influence of high power ultrasound and pasteurisation on the changes in the aroma profile and sensory attributes of apple juice and nectar. Results for the aromatic profile of juices showed that, compared to the untreated samples of juices and nectars, ultrasonic treatment lead to the formation of new compounds (which were not present in the untreated samples) or to the disappearance of compounds that were present in the untreated samples. Analysis of the aromatic profile of samples showed that the samples of juices and nectars after pasteurisation also had more aromatic compounds in comparison with the untreated samples. Esters are present at the highest fraction in both untreated and pasteurised apple juices and nectars.

Principal component analysis plot shows that some ultrasonically treated samples were different as sensed by electronic tongue compared to the untreated and pasteurised samples. There is also a qualitative difference between pasteurised samples and the untreated and ultrasonically treated samples. It can be concluded from the sensory evaluation that ultrasonically treated and pasteurised juices were evaluated with different scores in comparison with the untreated samples. The best accepted are ultrasonically treated apple juice (sample A1.10) treated at the amplitude of 60 μm and at 40 $^{\circ}\text{C}$ for 6 min and apple nectar (sample A2.6) treated at the amplitude of 120 μm and at 20 $^{\circ}\text{C}$ for 3 min. This work demonstrates that sonication influences the aroma profile, sensory properties and colour parameters of apple juices and nectars. Response surface methodology may be used to optimize critical process parameters to preserve the original properties of juices, which would make sonication an alternative technique to pasteurisation.

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