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EFFECT OF HIGH HYDROSTATIC PRESSURE TREATMENTS ON VOLATILES OF BERRY PURÉES

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High hydrostatic pressure (HHP) technology, as a promising alternative of thermal-treatment and chemical preservatives, can be used to produce minimally processed foods. It has the advantage of affecting only non-covalent bonds of macromolecules in foods, and thus preserves nutritional components, taste, and flavour exceptionally well. However, HHP also influences enzymatic reactions of food. Although some of these changes are often beneficial, monitoring the potential effects of high pressure treatments – especially in the field of product and technology development – is essential. The aim of this study was to point out some parameters of high hydrostatic pressure technique (pressure, temperature, build-up time, holding time, number of cycles) that can substantially impact the sensory properties of treated products.

Keywords: HHP, berry fruits, electronic nose, sensory analysis

High hydrostatic pressure technology (HHP), as a non-thermal preserving technique capable of inactivating or eliminating pathogenic and food spoilage microorganisms while retaining the valuable components of foods, has the potential to serve the needs of food-quality and food-safety simultaneously (Awuah et al., 2007).

HHP dates back over a century to the research of Bert Holmes Hite (1899) – who performed experiments with a variety of foods at elevated pressures –, albeit the technology seemed to be unremarkable until the end of the 1960s, when systematic researches started to explore its effects on microorganisms. Over the last 15–20 years, significant advances took place in the technology, mainly because high pressure equipments have become commercially available (Patterson et al., 2007). Nowadays, HHP technique inspires dozens of manufacturers to produce innovative, natural-looking, fresh-like foods that satisfy consumers' need (Oey et al., 2008).

When using HHP treatment, an elevated pressure is applied in an instantaneous way throughout the product, subjecting foods to 100–1000 MPa under water or a special fluid as pressure transmitting medium (Cao et al., 2012). Due to the instantaneously transmitted pressure, processing time and conditions are independent of the volume and shape of the treated sample (Norton & Sun, 2008; Karim, 2011).

Beside the inactivation of microorganisms, there are some further effects of pressure on foods. Without the claim of completeness, these are related to enzyme activation or inactivation, protein denaturation and modification, gel formation, not mentioning the changes in the properties of carbohydrates and fats. Although it is generally assumed that the flavour of foods is not impaired by high pressure – since the structure of small volatile compounds is not affected –, HHP processing can induce some enzymatic and chemical

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reactions that finally cause changes in flavour, too. To draw proper conclusions, there is an unequivocal need to integrate objective measurement tools into the generally non-objective sensory ways. Accordingly, it is useful and scientifically justified to apply “artificial sensory tools” when analysing the objective attributes of treated samples.

Electronic nose offers a fast and non-destructive way to sense aroma, so it can be prosperously used to identify the numerous volatile compounds of foods. However, when examining samples with electronic nose, a complex pattern is created without the possibility of defining the aroma components individually. Nowadays, beside the challenges in food authenticity assessment, food quality control, and shelf-life investigation, “e-nose” can be also applied to evaluate food freshness and to reveal changes caused by some preservation techniques (Wilson & Baietto, 2009).

The aim of this study was to determine some parameters of high hydrostatic pressure treatment that can significantly modify the volatiles of berry purées.

1. Materials and methods

1.1. Samples

Strawberry and raspberry purées were produced with a Robot-Coupe C80 type automatic sieve (Robot-Coupe Ltd., Montceau-les-Mines, France) and used as raw samples, which had been portioned into small polyethylene pouches and heat-sealed airtightly. Plastic pouches were frosted with a Nortech QCF 103 blast chiller (Normann Srl., Orsago, Italy) and then put under frozen storage at –24 °C.

1.2. HHP treatments

Following thawing, HHP treatments were carried out in a Resato FPU-100-2000 HHP equipment (Resato Int. B.V., Roden, Netherlands) that contained a pressurizing (1600 mm × 2200 mm × 830 mm) and a control (1300 mm × 950 mm × 1400 mm) unit. The capacity of the pressurizing chamber was 2 litres, and a so called Resato PG Fluid served as pressure transmitting medium. Where higher temperatures were used, samples were chilled in icy water just after treatments.

Table 1 summarizes the parameters of each HHP treatment. The “build-up time” column means the interval needed to reach 100 MPa pressure elevation. Values represented in the table were mainly based on the settings of industry-wide applied HHP treatments.

1.3. Measurements with electronic nose

Electronic nose measurements were performed with an NST 3320 type instrument (Applied Sensor, Linköping, Sweden), which had a built-in headspace sampler for 12 samples, a detector unit containing 23 different sensors, and a software for collecting and processing the data stem from the sensors. NST 3320 consisted of 10 metal oxide semiconductor field effect transistor (MOSFET) sensors, 12 metal oxide semiconductor (MOS) sensors, and a sensor for the detection of relative humidity. The response of the MOS sensors is measured as the change in resistance between the electrodes as a result of the chemical reactions occurring at the surface of the metal oxide semiconductor, while MOSFET sensors are based on a change in the electrostatic potential. Ambient air was filtered through a silica gel drying column, and a combined moisture/hydrocarbon filter was used as clean reference gas for the sensors. The gas flow rate of the dynamic sampling was set to 50 ml min^{-1}.

Table 1. Parameters of high hydrostatic pressure treatment

	Pressure (MPa)	Tempera-ture (°C)	Build-up time (sec)	Holding time (min)	Number of cycles (pcs)
Treat-ment 1.	400 500 600	10	60	5	1 1 treatment cycle with a 5 min – 300 sec – long holding time
Treat-ment 2.	400	10 30 50	60	5	1 1 treatment cycle with a 5 min – 300 sec – long holding time
Treat-ment 3.	400	10	60 120 180	5	1 1 treatment cycle with a 5 min – 300 sec – long holding time
Treat-ment 4.	400	10	60	5 10 15	1 1 treatment cycle with a 5 min – 300 sec – long holding time
Treat-ment 5.	400	10	60	5	1 1 treatment cycle with a 5 min – 300 sec – long holding time 2 2 treatment cycles, each with a 2.5 min – 150 sec – long holding time 3 3 treatment cycles, each with a 1.67 min – 100 sec – long holding time

Control (untreated) and HHP-treated purées were put into special glass vials of the electronic nose and were closed by Teflon coated septa. Five grams of sample were put into each vial. Measurements were carried out at stock settings of the e-nose. These settings were framed of a 20 °C incubation temperature, 20 min incubation time, 30 sec sampling time, 60 sec flushing time, and a 260 sec long regeneration time. The sequences of the vials were allocated by using a random number generators. The instrument examined each vial in three repetitions. Difference of sensor signals between the baseline and the signal value at the end of the sampling time was used for multivariate statistical analysis as sensor response.

1.4. Sensory analysis

Sensory analysis was performed by an untrained sensory panel – with 12 attendants in average – to compare the ability of the e-nose and human perception to differentiate between control and treated berry purées. The panel evaluated the samples subjected to different HHP treatments by using a triangle test (Lawless & Heymann, 2010), where members were also asked to mark their judgements with a reliability index.

1.5. Data analysis

During the experiments Linear Discriminant Analysis (LDA) (SPSS 20.0 for Windows, Chicago, Illinois, USA) was applied to obtain classification rules for differentiation between berry samples when evaluating electronic nose measurement data.

2. Results and discussion

Figure 1 shows the first two typical examples of discriminant analysis results, which are calculated from the sensor responses of the e-nose when different pressure levels were applied for treating raspberry and strawberry purée samples. Based on the distance of different groups, similarity of the classes can be evaluated. Closer groups mean less change in volatiles. As can be seen, control groups separated well from treated samples when raspberry purées were examined, however, the distance of untreated and HHP-treated groups shrank when strawberry samples were put under investigation. These observations were confirmed by the outputs of cross-validation, where a higher classification efficiency could be reached amongst raspberry samples. For example, the control and 600 MPa groups of raspberry purées could be identified 88.9% and 77.8% correctly, respectively. At the same time, these numbers were far weaker when examining strawberry samples: 55.6% and 33.3%.

Nevertheless, the distances between treated samples were smaller than between control and treated ones, and a clear trend could also be realised in the location of treated sample groups. Application of a higher treatment level resulted in a bigger distance from the control group.

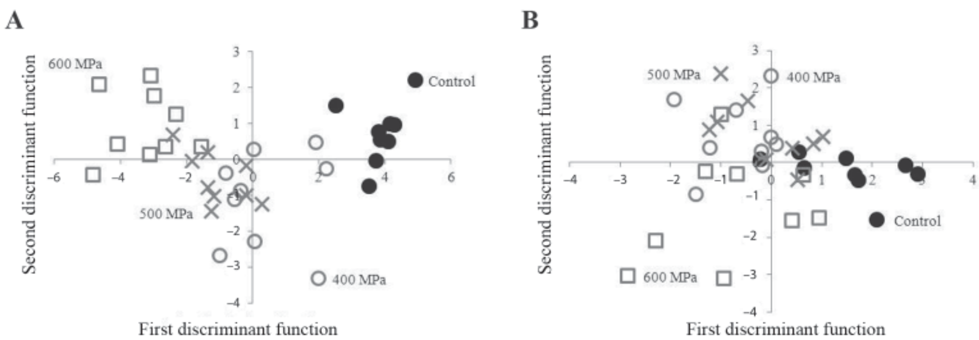

Fig. 1. Discriminant analysis score plots of raspberry (left) and strawberry (right) purées treated with high hydrostatic pressure at different pressure levels – based on the sensor-responses of electronic nose. (●: Control; ○: 400 MPa; ×: 500 MPa; □: 600 MPa). A: Raspberry; B: strawberry

As Fig. 2 shows, the different temperatures resulted in a respectable disjunction of control and HHP-treated berry purées. Although the individual samples of the 50 °C strawberry group demonstrated a considerably bigger deviation, control samples could be separated 100% correctly from the HHP-treated ones in both cases.

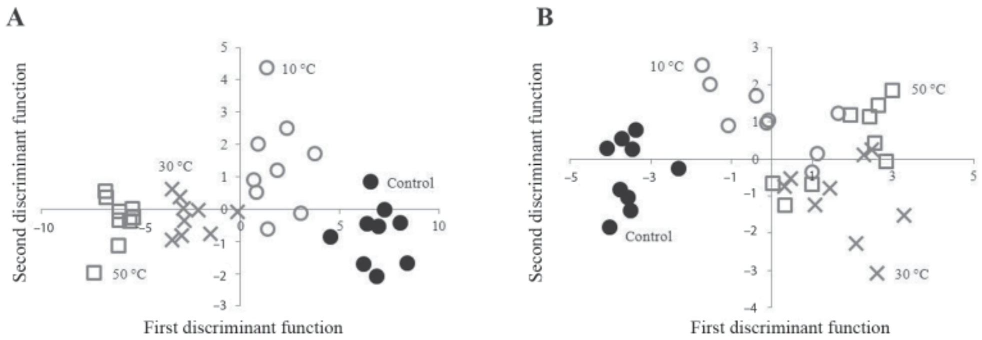

Fig. 2. Discriminant analysis score plots of raspberry (left) and strawberry (right) purées treated with high hydrostatic pressure at different temperatures – based on the sensor-responses of electronic nose.
(●: Control; ○: 10 °C; ×: 30 °C; □: 50 °C)); A: raspberry; B: strawberry

Figure 3 clearly points to the fact that raspberry and strawberry groups showed different tendencies when applying various holding times. Even though control samples could be divided from the treated purée groups in every case, a noticeable overlap appeared between other HHP-treated subgroups. The results of cross-validation verified that the efficiency of classification was weaker among strawberry samples again, not surprisingly.

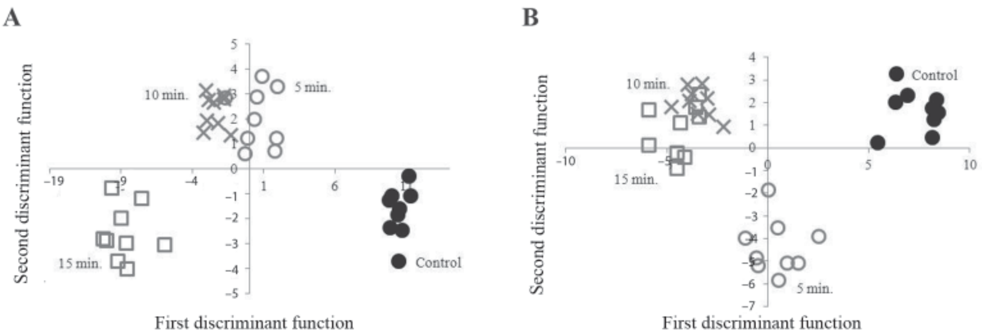

Fig. 3. Discriminant analysis score plots of raspberry purées treated with high hydrostatic pressure at different holding times – based on the sensor-responses of electronic nose.
(●: Control; ○: 5 min; ×: 10 min; □: 15 min);). A: Raspberry; B: strawberry

Table 2 summarizes the main indices related to the efficiency of classification for both berry fruits. Eigenvalues are also represented in the table, which intrinsically compare the deviation measured between and within groups. The bigger the Eigenvalue, the better the groups can be divided from each other. As represented in every Figure above, the greatest difference between groups appeared along the first function, so Table 2 contains the Eigenvalues of the first discriminant function only. Values marked by superscript „a” or „b” show the cases of the best and the second-best classifications, respectively.

Table 2. Effects of different HHP parameters on the typical indices of discriminant analysis based on the sensor-responses of the electronic nose

Modified parameter of HHP treatment	Eigenvalue of the first discriminant function		Efficiency of classification (%)			
			Original model		Cross-validated	
	Raspberry	Strawberry	Raspberry	Strawberry	Raspberry	Strawberry
Pressure	7.554	0.922	88.9	66.7	58.3	33.3
Temperature	26.32	5.38 [b]	100 [a]	80.6 [b]	86.1 [a]	63.9 [b]
Build-up time	28.957	3.302	97.2 [b]	77.8	52.8	38.9
Holding time	54.109 [b]	24.221 [a]	97.2 [b]	100 [a]	86.1 [a]	72.2 [a]
Number of cycles	55.256 [a]	1.445	94.4	69.4	75 [b]	41.7

Superscript "a" and "b" mean the highest and the second-highest values related to the best and the second-best classification

Table 2 demonstrates that different holding times had the greatest effects on the discrimination of the groups, and temperature had the second greatest. It can be seen that different pressures had minor effects regarding the discrimination of groups.

Results of the sensory analysis (Table 3) showed that attendants were not able to separate the control group from the treated samples in general. Even where correct answers were given – based on the marked reliability indexes −, choices could not be attributed to solid considerations. Only the raspberry purées treated with the highest temperature (marked by grey) proved to be significantly different from other samples.

Table 3. Results of triangle tests when samples treated with different parameters of HHP were put under sensory analysis

Modified parameter of HHP treatment	Determined levels (low-medium-high)	Number of correct answers						Number of panellists	Minimum number of correct judgments to establish significance at probability level of 5%
		Raspberry			Strawberry				
		low	medium	high	low	medium	high		
Pressure (MPa)	400-500-600	3	5	4	2	3	1	11	7
Temperature (°C)	10-30-50	4	3	7	5	5	3	10	7
Build-up time (sec/100 MPa)	60-120-180	3	3	3	6	6	5	13	8
Holding time (min)	5-10-15	3	5	4	2	6	2	17	10
Number of cycles (pcs)	1-2-3	1	2	3	4	3	3	10	7

3. Conclusions

According to our study, the sensors of the electronic nose were capable of distinguishing the subgroups of HHP-treated samples from the untreated ones.

Results confirmed that the potential effects of high pressure treatment – combined with mild heat – on the volatiles of berry purées cannot be certainly prognosticated. For example, when samples were subjected to different pressures, treated groups of raspberry purées definitely separated from the control samples. At the same time, the difference diminished between strawberry groups where bigger overlaps appeared and the deviation of the individual samples within groups also increased. Former studies came to similar results, when heat- and HHP-treated raspberry, strawberry, and blackcurrant purées were examined with e-nose, just after treatments as well as during two- and four-week long storage times (Dalmadi et al., 2007; Dalmadi, 2009).

Comparing the effects of the parameters, it could be stated that holding time and temperature principally affected the volatile compounds, while the impact of different pressures was not so remarkable.

Furthermore, experiments confirmed that the human sensory panel could not differentiate between raw (control) and treated samples. Thus, it could be conceived that e-nose proved to be a better tool when classifying control and HHP-treated berry purées.

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