Changes during the Extrusion of Semolina in Mixture with Sugars

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Abstract

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Wheat semolina and its mixtures with 5% glucose, fructose of sucrose were processed in a sigle screw extruder at the maximum temperature of 140°C and the processing time of 30 s. The nonenzymic browning was only moderate, but it was substantially more intensive in mixtures with glucose or fructose than in the case of wheat semolina or its mixture with sucrose. Red and yellow pigments were mainly formed. The odour acceptability was affected by the presence of sugars almost negligibly, but the intensities were different, higher in extruded mixtures with glucose and fructose than in wheat semolina or its mixture with sucrose. Small differences were observed in the sensory profile. Extrusion of semolina with sugars produced more sensory active volatiles (52–69 identified compounds) than in extruded semolina (41 compounds). Pyrazines, furans and pyrans were the most important sensory active compounds. Their amounts increased by the addition of sugars to semolina; the mixture of semolina with glucose was particularly rich in active compounds. The formation of pyrazines was more enhanced by the addition of fructose than of other sugars. Maltol, butyrolactone and acetic acid were present in large amounts. Even if sensory characteristics were improved by addition of sugars to semolina, the difference was not very pronounced.

Keywords: browning; extrusion; furans; fructose; glucose; pyrazines; semolina; sensory value; sucrose; wheat

Extrusion cooking has become a favourite alternative to baking, particularly for the technological processing of cereals and other starchy foods into a variety of food products (HUBER 1991). The main advantage is short processing time, low energy input, and lower costs, compared to baking.

Naturally, wheat flour, semolina or grits are, the most widely used cereal raw material, and various extruded products may be obtained with good reproducibility as the process is continuous (LUNDGREN *et al.* 1991/2). Triticale was also found advantageous for extruded products because of interesting flavour notes formed, compared with wheat (PFANNHAUSER 1993). The process was optimised for whole triticale kernels (LORENZ *et al.* 1974). Uncooked or pregelled starches (or starch-rich wheat fractions obtained by air classification) can be also used instead of flour, especially for snack foods (FELDBERG 1969).

Another important raw material for extrusion cooking is corn meal, which is transformed into extrudates of interesting flavour character (CHEN *et al.* 1991). However, the main advantage of corn processing is time, energy and cost saving, similarly as in wheat products (MIDDEN 1989). Corn meal can be mixed with other ingredients, such as amaranth or rice (JOTOVALLOTOVAL & SEIBEL 1992), in order to improve sensory and functional properties of the product. Extruded rice flakes are fully acceptable for breakfast cereals (VISSESURAKARN *et al.* 1991).

A disadvantage of extruded products is their weaker aroma and flavour intensity than that of bread crust or of other traditional bakery products. Therefore, the enhancement of reactions resulting in the formation of sensory active substances is very important. Pyrolysis of proteins, caramelisation of sugars, Maillard and related reactions should be taken into account (OSNABRUGGE 1981). Furan derivatives and maltol originate from sugar degradation during extrusion cooking (RIHA & HO 1996), which are similar to those found in bread aroma (ROTHE 1974). They are present in large amounts, but nitrogen-containing heterocycles are more important because of their very low perception thresholds (BOELENS & HEYDEL 1973). Pyrazines were isolated from bread crust (ROTHE 1974) and bread crumb (SCHIEBERLE & GROSCH 1984). They are also important in other extruded cereal products, such as corn tortillas (KARAHADIAN & JOHNSON 1993; BUTTERY & LING 1995). Their most probable pathway of formation are interactions of Maillard and Strecker degradation products (RIHA & HO 1996; PFANNHAUSER 1993; YOO & HO 1997). Their precursors are mainly free amino acids (HWANG *et al.* 1995), but they are also produced by interactions of saccharides with protein, for instance in the extrusion cooking of potato flakes (MAGA & SIZER 1979). Another pathway is probably based on the formation of dihydropyrazines, and their subsequent oxidation to pyrazines *via* the respective free radicals (MILIĆ *et al.* 1980).

The aroma intensity of extruded products may be increased by the fortification of the material to be extruded with reducing sugars and amino acids, which are the most important precursors of Maillard reactions. Honey belongs to highly appreciated sources of reducing sugars. Addition of honey to whole meal increased the sensory value of extruded breakfast cereals (NEUMANN & CHAMBERS 1993). Malt, which contains low molecular-weight hydrolytic products of starch, is another suitable ingredient, increasing the formation of pyrazines (FORS 1987). Maltose, glucose or fructose syrups would be a good choice, too.

In this paper we compare the effect of glucose, fructose and sucrose on the flavour enhancement of extruded wheat semolina.

MATERIAL AND METHODS

Material: Wheat semolina type T 600 (following the Czechoslovak standard – ČSN) was produced in agrement with the respective national standard; it contained 12.4% moisture, 0.4% ash, 8.6% protein and 0.3% fat. Sucrose, D-glucose and D-fructose were purchased from Sigma-Aldrich (St. Louis, MN, USA). Sugars were ground separately in a Moulinex mill (Moulinex, Paris, France), and 5.0 kg of wheat semolina was then blended with 250 g of finely ground saccharides in a drum-type two-roller mill (Romil, Brno, CR), provided with grooving rolls. The material was air dried (20°C, 50% relative humidity) before the extrusion.

***Extrusion Process*:** The material (5.0 kg of semolina and 0.25 kg of the respective saccharide) were thoroughly mixed, and then fed into the extruder. The samples were extruded under the conditions summarized in Table 1, in a pilot plant-scale equipment, manufactured by the Research Institute of Milling and Baking Industry, Machinery Department (Prague, CR). The extruded samples were crushed immediately, and stored at room temperature in ground glass bottles.

Extraction of Volatiles: The Solid Phase MicroExtraction (SPME) procedure was used. A 65 µm Carbowax™ divinylbenzene fibre for Manual Holder (Red label) was produced by Supelco (Bellefonte, USA). The extraction time was 1 h at 85°C, and the desorption time was 2 min at 220°C. The fibre was cleaned for 30 min at 220°C before extracting the next sample.

Gas Chromatography: For gas chromatography (GC), an apparatus GC 8000 (Fisons Instruments, Milan, Italy) was equipped with an autosampler HS 8000; injection temperature: 220°C; column: Supelco 60 m × 0.32 mm, coated with Supelcowax 10, film thickness: 0.25 µm (Supelco, Bellefonte, USA); column temperature programming: 50°C for 2 min, isothermal, then heating by 2 K/min to 220°C, and isothermally for 30 min at 220°C; carrier gas: helium; initial pressure 100 kPa; the inject:split ratio is 1:25; FID detector; detector temperature 220°C.

Table 1. Conditions of extrusion cooking

Parameter	Experimental value
Extruder	Single screw collet extruder VUMPP 83
Type of screw	3-way
Distance between flights	36 mm
Screw rotation	5.85 Hz
Dosing	1.923
Feed rate	only in the feed zone
Residence time	40 kg/h
Maximum extrusion temperature	30 s
Dosing	140°C
Shaping dies	12 (diameter
Distance dies	88 (diameter
Die temperture	110°C

Mass Spectrometric Detection: For GC with mass spectrometric detection (GC/MS), the Fisons MSD 8000 mass spectrometer was used; the Manual SPME Injection Method was applied; the ionizing energy was 70 eV. Identifications were based on the comparison with the MS Computer Library (NIST-Masslab-Software Package, Fisons, Milan, Italy), and on the respective retention indices.

Determination of Browning Degree: Samples of extrudates were finely ground for 30 s in a Moulinex processor (Moulinex, Paris, France). The colour hue of the ground samples was measured using a CCD Fiber Optic Spectrometer S 200 (manufactured by Ocean Optic, Ltd., Dunedin, FL, USA). The reflected light was measured at the constant luminance (L*) of 50%. The colour hue was expressed in the CIE-LAB space, using a modular spectroscopy system in the reflectance mode. A standard light source D 65 after Comité International de l'Eclairage (CIE) was used, corresponding to the sun surface (6500 K). The reflection assembly probe consisted of 7 fibres in a ferrule. The results were processed by the software Spectrawin Version 3.1, and the following CIE-LAB parameters were determined: the a* value – redness (positive) to greenness (negative); the b* value – yellowness (positive) to blueness (negative); the hue angle: $h^* = \tan^{-1} b^*/a^*$; the chroma: $c^* = (a^{*2} + b^{*2})^{0.5}$; the hue difference $\Delta E = (\Delta a^{*2} + \Delta b^{*2} + \Delta L^{*2})^{0.5}$.

Sensory Analysis: Conditions for the sensory analysis were in agreement with specifications of the international standard (ISO 6658-1985: Sensory analysis – Methodology – General guidance); test rooms (ISO 8589-1989: Sensory analysis – General guidance for the design of test rooms); selected, trained and monitored assessors (ISO 8586-1991: Sensory analysis – General guidance for the selection, training and monitoring of assessors, Part 1: Selected assessors); unstructured graphical scales (ISO 4121-1978: Sensory analysis – Grading of food products by methods using scale categories) were represented by straight lines 70 mm long, provided with descriptions on either end for assessor's orientation (intensity of brown colouration: 0 mm = very weak, 70 mm very strong; odour acceptability: 0 mm = very disagreeable, 70 mm = very agreeable; odour intensity: 0 mm = very weak, 70 mm = very strong; texture acceptability, as assessed by pressing small amount of coarse extruded grits between fingers: 0 mm = pasty, 70 mm = very crispy). The sensory profile (ISO 6564-1995: Sensory analysis – Flavour profile) was based on free choice profiling, and 32 descriptors were originally collected, which were compressed into 10 more complex descriptors: 1 = roasted, bread crust, roasted peanuts, gingerbread with honey; 2 = burnt, caramel, bitter; 3 = woody, bark, resins; 4 = pasty, stored flour, stale, bread crumb; 5 = spicy, onion, garlic, sulphuric; 6 = sharp, pungent, burning; 7 = fatty, oily, buttery; 8 = earthy, musty, mouldy, sweat, wet dog; 8 = malty, cocoa, sweet; 9 = solvents, synthetic, chemicals; 10 = others – specify, which; in the odour profile evaluation, unstructured graphical scales were again used: 0 mm = odour note absent, imperceptible, 70 mm = very strong. The colour intensity (degree of browning) was assessed under the standard light source C (specified by CIE, corresponding to the spectrum of sun surface = 6500 K). Odour profiles were tested by sniffing from a ground wide-neck glass bottle, 250 ml volume. The results are based on 20 responses (standard deviations of the means were 3–6% of the graphical scale).

RESULTS AND DISCUSSION

The following extruded samples were prepared, and analyzed:
A = extruded semolina;
B = extruded mixture of semolina + 5 % D-glucose;
C = extruded mixture of semolina + 5 % D-fructose;
D = extruded mixture of semolina + 5 % sucrose.

All samples were analysed within 2–3 weeks after preparation, and were stored in a refrigerator in ground-glass bottles in the meantime.

Evaluation of Browning Degree: The effects of sugars on the browning degree during the extrusion are evident from Table 2. The degree of browning was small in the case of semolina without sugar additions (sample A) or with the addition of sucrose (sample D), which belongs to non-reducing sugars, and may react only after hydrolytical cleavage into a mixture of glucose and fructose. The browning of mixtures of semolina with reducing sugars, such as glucose or fructose (samples B and C, respectively) was substantially more intensive. The same applies of different CIE-LAB characteristics. The pronounced increase was observed after the addition of glucose or fructose to semolina, slightly more intensive in mixture with glucose than in a mixture with fructose. It may be due to differences in reaction rates of browning intermediates between Amadori (Pokorný *et al.* 1988) and Heyns (Pilková *et al.* 1990) rearrangements. The increase of a* and b* corresponds to the increase in redness and yellowness, respectively, which occurs in the beginning of nonenzymic browning. Products with more intensive green and blue colour notes are formed only later, as a result of secondary reactions. They cannot occur in course of very short extrusion time of 30 s, and at relatively low temperatures (120–140°C).

Odour (Aroma) Evaluation of Extruded Products: The odour acceptability was not significantly affected by the presence of sugars in the extruded mixture, added before

Table 2. Colour parameters of extruded samples

Characteristic	Sample A	Sample B	Sample C	Sample D
Sensory colour intensity [mm]	17.2	40.5	30.2	14.6
a* value	11.95	28.39	24.02	15.05
b* value	64.79	76.78	74.28	66.94
h* angle	79.49	69.89	72.29	76.67
Chroma C*	64.24	81.80	77.80	69.30
Colour difference ΔE	59.49	77.35	73.09	62.64

Table 3. Sensory odour and texture evaluation of extruded samples (values are expressed in mm of the graphical scale)

Sensory characteristic	Sample			
	A	B	C	D
Texture acceptability	47.3	42.6	43.3	48.2
Odour acceptability	38.6	36.4	38.1	39.8
Odour intensity	19.6	24.7	21.7	19.4
Sensory odour profile				
Roasted, bread crust	22.1	27.4	27.0	23.5
Caramel, burnt	14.4	19.5	28.5	24.2
Pasty, bread crumb	40.0	33.2	44.2	27.8
Spicy	3.9	2.1	7.8	5.8
Fatty	6.6	8.8	10.4	10.7
Earthy	4.2	3.9	5.7	3.2
Sharp, sweat, pungent	5.5	3.7	9.6	3.2

the extrusion, obviously because of similar roasted character, which is perceived as the main, and very positive sensory attribute (Table 3). The odour intensity was somewhat higher in extruded mixtures of semolina with reducing sugars, whereas no effect was observed in the case of a mixture with sucrose.

The texture, expressed as crispness, varied only a little so that insignificant differences were observed by sensory analysis. It is an advantage for the comparison of samples, as low crispness has an adverse effect on the sensory quality not only of texture, but also of aroma and flavour (STREJČEK *et al.* 1993).

The flavour profile depended on sugars added to the extruded mixture. The presence of reducing sugars (samples B and C) increased the roasted character, typical for bread crust, but sucrose (sample D) had no effect. All the three sugars increased the intensity of caramel-like odour note, which is similar to burnt odour (Table 3). At the same time, the intensity of fatty, buttery odour note increased, too. Solvent, chemical or malty odour notes were so weak (less than 5 mm of the graphical scale in the average), that they have not been included in the Table 3. Large differences were observed in the intensity of a flavour note after stored flour, paste or bread crumb.

Volatile Sensory Active Compounds in Extruded Samples: Examples of gas chromatographic analysis of volatiles from the samples A–D are shown in Figs. 1–4, respectively.

The list of detected and identified compounds of the volatile fractions is shown in Table 4. Only 41 compounds were identified in extruded semolina without any additions (sample A, Fig. 1); of course, much higher number of compounds would be produced by thermolysis at higher temperature (ANDREJS *et al.* 1995), such as during baking. In all samples, furans and pyrazines belonged to the most numerous substances.

Table 4. Number of substances identified in the volatile fraction

Compounds class	Sample			
	A	B	C	D
Pyrazines	11	16	17	16
Furans, pyranes	5	14	12	8
Pyrroles	3	4	2	3
Sulphur heterocycles	1	0	0	1
Carbocyclics	3	7	2	2
Aldehydes	4	7	5	5
Ketones	3	8	3	3
Alcohols	2	3	3	3
Fatty acids	6	7	5	5
Hydrocarbons	3	3	5	6
Total	41	69	54	52

The total number of compounds was higher in extruded mixtures with sugars (samples B, C and D, Figs. 2–4, respectively) than in extruded semolina, including furans and pyrazines. From this respect, the composition of volatiles was similar to that of bread crust, even when the overall intensity was weaker. At very high temperatures and higher content of precursors, the number of furan derivatives was reported to be much higher (BALTES & BOCHMANN 1987a). During bread baking, the temperature and duration of heating are substantially higher than during extrusion cooking. The pyrrole and pyridine fractions therefore become more important in bread crust. No pyridines were detected in our extruded samples.

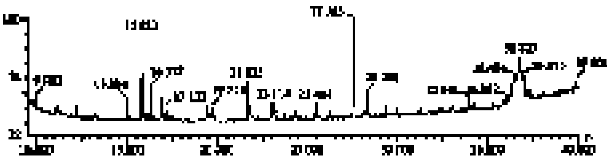

Fig. 1. Gas chromatogramme of volatiles isolated from extruded semolina (Sample A)

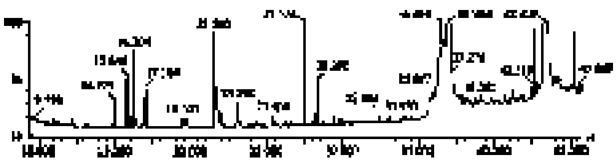

Fig. 3. Gas chromatogramme of volatiles isolated from extruded mixture of semolina with fructose (Sample C)

Axis *y* – detector response [p. c. of the scale]; Axis *x* – retention time [min]; the respective retention times are attached to individual peaks

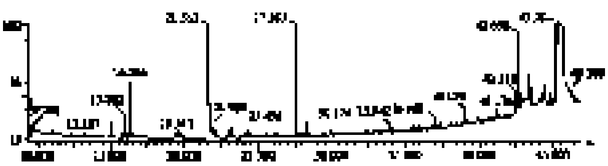

Fig. 2. Gas chromatogramme of volatiles isolated from extruded mixture of semolina with glucose (Sample B)

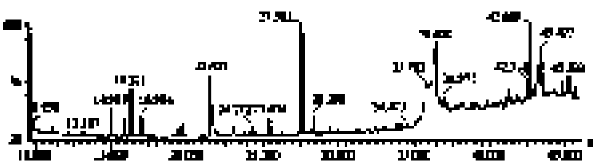

Fig. 4. Gas chromatogramme of volatiles isolated from extruded mixture of semolina with sucrose (Sample D)

Table 5. Contents of classes of sensory active volatiles (expressed in p.c. of total peak areas)

Compounds class	Sample			
	A	B	C	D
Pyrazines	2.65	5.62	13.3	4.79
Pyrroles	0.08	0.80	0.30	0.22
Furans	0.78	9.04	7.37	
Butyrolactone	12.64	20.40	24.31	2.24
Maltol	0	14.95	9.88	19.09
Benzaldehyde	0.39	0.23	0.35	8.07
Alkanals	10.98	4.92	6.09	0.57
Ketones	3.87	7.82	7.70	6.02
Alcohols	2.28	4.08	3.48	10.13
Volatile fatty acids	34.28	23.35	18.05	32.17

The quantitative distribution of different classes of compounds is evident from Table 5. The content of pyrazines, pyrroles and furans in the volatile fraction was increased by the addition of sugars, which is not surprising in the case of furans, as they are produced from sugars. The content of other precursors, protein and free amino acids, decreased following the addition of sugars, but the decrease was more than compensated by a higher concentration of other precursors – namely the decomposition products of sugars, so that the resulting fraction of pyrazines was much larger. Proteins, peptides and amino acids, present as natural components in semolina, were obviously sufficient for the formation of pyrazines. The amount of ammonia, necessary for the pyrazine formation, can be liberated not only by the cleavage of the peptide bond, but also by deamidation of glutamine and asparagine bound in proteins (ZHANG 1994).

The amount of aldehydes and various dicarbonylic precursors increased by decomposition of sugars, e.g. the addition of glucose enhanced the formation of 2,3-pentadione and 1-hydroxy-2-propanol, which became a major product (6.4–7.6% total volatiles). Biacetyl was not detected because of its high volatility, but 2,3-butandiol was higher by 1–2% of total volatiles in extruded mixtures with sugars than in extruded semolina. The content of butyrolactone substantially increased, too. No maltol was detected in extruded semolina (sample A), but it became a major compound in extruded mixtures with sugars. Maltol is produced by thermolysis of Amadori compounds (YAYLAYAN *et al.* 1992). Naturally, there were much more abundant in extruded mixtures of semolina with sugars (samples B–D). On the contrary, the content of benzaldehyde, which does not belong to Maillard products, was nearly the same in all samples; it contributes to the moderately bitter odour of extruded samples.

Alkanals detected among the volatiles could not have much pronounced influence on the resulting flavour as they were high molecular-weight compounds (10–12 carbon atoms). Fatty acids, mainly acetic acid, were also not substantial as flavour carriers. Hydrocarbons with 10–18 carbon atoms were present, but they cannot be considered as substances of major importance because of their higher detection thresholds.

Table 6. Contents of individual pyrazines in extruded samples (expressed in p.c. of peak areas of the total pyrazine fraction)

Pyrazine substituents	Sample			
	A	B	C	D
Unsubstituted	8.3	12.1	1.9	6.9
Methyl	24.2	32.9	24.6	30.5
2,5-Dimethyl	21.5	2.5	19.1	16.5
2,6-Dimethyl	13.6	6.2	26.8	14.8
Ethyl	0	7.1	1.0	2.9
2,3-Dimethyl	1.9	2.1	1.6	3.1
2-Ethyl-6-methyl	1.1	1.2	1.4	1.7
2-Ethyl-5-methyl	0.8	1.1	1.4	1.5
Trimethyl	15.1	22.8	13.0	18.2
Vinyl	0	1.8	0.8	1.5
2-Ethyl-3,5-dimethyl	2.4	0.5	1.5	1.7
2-Vinyl-6-methyl	0	0.2	0.6	0.4
2-Vinyl-5-methyl	0	<0.2	1.9	0.2
Acetyl	0	0.2	0.1	0.2
5-Methyl-2-acetyl	2.3	2.3	1.5	0.2
6-Methyl-2-acetyl	9.1	6.8	0.7	0.2
2-Hydroxypropyl	0	0	2.2	0

In the discussion on the composition of volatiles it should be made clear that the results depend on the extraction method. In the case of the SPME method it depends mainly on properties of the film on the surface of the fibre. The conclusions thus apply to the Carbowax fibre; the affinity of volatiles to smell receptors may be still different, anyway, so that the results should be always compared with sensory characteristics.

The most sensory active group of extruded volatiles – pyrazines, were influenced by sugar addition not only in their total amount, but also in the composition. Relative concentrations of individual pyrazines (expressed in p.c. of peak area of total pyrazines) are shown in Table 6. The composition of volatile pyrazines also depends on the intensity of heating. Relatively low contents of trimethyl pyrazine and 2,6-dimethyl-3-ethyl pyrazine were observed in wheat bread crust, which was exposed to high temperatures over 200°C (SCHIEBERLE & GROSCH 1987), whereas relatively high amounts were present in our extruded samples, heated to only 140°C. Pyrazines identified in our volatiles mostly consisted of methyl and methyl-ethyl substituted derivatives. They were reported among pyrazines from crust of white American bread (SIZER *et al.* 1975). Traces of ethyl, vinyl and acetyl derivatives were only detected in extruded samples containing sugars (samples B–D), whereas ethylmethyl and methylacetyl derivatives were present even in extruded semolina (sample A). It is in agreement with the formation of both 2-vinyl-5-methyl pyrazine and 2-vinyl-6-methyl pyrazine in extruded malt (FORS & ERICSSON 1986), which also contains reducing sugars. Pyrrolopyrazines and furanopyrazines were not found, although they were detected in roasted model samples (BALTES & BOCHMANN 1987b), as in our extrusion experiments, the temperature was too low and the content of free amino acids negligible. Cyclopentapyrazines were also not found from the same reason.

CONCLUSIONS

The addition of sugars to semolina moderately improved the colour and sensory characteristics of extruded products, mainly as a result of increased amounts of pyrazines and furans. However, the differences between extruded semolina and extruded mixtures of semolina with sugars were not very pronounced.

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Souhrn

FAROUK MANSOUR A., PUDIL F., JANDA V., POKORNÝ J. (2001): **Změny pšeničné krupice ve směsi s cukry během extruze**. Czech J. Food Sci., **19**: 24–30.

Pšeničná krupice a její směsi s 5 % glukosy, fruktosy nebo sacharosy byly zpracovány na jednošnekovém extrudéru při maximální teplotě 140 °C a prodlevě 30 s. Za těchto podmínek proběhlo neenzymové hnědnutí v malé míře u krupice nebo její směsi se sacharosou, znatelnější bylo u směsí s glukosou nebo fruktosou (vznikaly hlavně žlutě až červeně zbarvené produkty). Textura byla u všech vzorků podobná. Cukry měly zanedbatelný vliv na příjemnost chuti, ale intenzita vůně byla průkazně vyšší u směsí s glukosou nebo fruktosou než u směsi se sacharosou nebo u krupice samotné. Byly také zjištěny rozdíly v senzorických profilech vůně – u extrudovaných produktů s cukry bylo prokázáno více těkavých látek (52–69 identifikovaných) než u extrudované krupice (41 látek). K senzoricky nejvýznamnějším sloučeninám patřily pyraziny, furany a pyrany, jejichž počet i množství po přídavku cukrů vzrostly (po přídavku glukosy hlavně u furanů a fruktosy u pyrazinů). K dalším význačně zastoupeným produktům patřil butyrolakton, maltol a kyselina octová. I přes mírné zlepšení senzorických charakteristik se vliv cukrů projevil málo výrazně.

Klíčová slova: extruze; furany; fruktosa; glukosa; hnědnutí; krupice; pšenice; pyraziny; sacharosa; senzorické vlastnosti

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