

Identification of Potent Odorants Formed during the Preparation of Extruded Potato Snacks

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Extrusion cooking processing followed by air-drying has been applied to obtain low-fat potato snacks. Optimal parameters were developed for a dough recipe. Dough contained apart from potato granules 7% of canola oil, 1% of salt, 1% of baking powder, 5% of maltodextrin, and 15% of wheat flour. After the extrusion process, snacks were dried at 85 °C for 15 min followed by 130 °C for 45 min. The potent odorants of extruded potato snacks were identified using aroma extract dilution analysis and gas chromatography–olfactometry. Among the characteristic compounds, methional with boiled potato flavor, benzenemethanethiol with pepper-seed flavor, 2-acetyl-1-pyrroline with popcorn flavor, benzacetaldehyde with strong flowery flavor, butanal with rancid flavor, and 2-acetylpyrazine with roasty flavor were considered to be the main contributors to the aroma of extruded potato snacks. Several compounds were concluded to be developed during extrusion cooking, such as ethanol, 3-methylbutanal, (*Z*)-1,5-octadien-3-one with geranium flavor, and unknown ones with the flavor of boiled potato, cumin, candy, or parsley root. Compounds such as methanethiol, 2,3-pentanedione, limonene, 2-acetylpyrazine, 2-ethyl-3,5-dimethylpyrazine, 4-hydroxy-2,5-dimethyl-3(*2H*)-furanone, 3-hydroxy-4,5-dimethyl-2(*5H*)-furanone, 2-methyl-3,5-diethylpyrazine, 5-methyl-2,3-diethylpyrazine, and (*E*)- β -damascenone were probably developed during air-drying of the potato extrudate.

KEYWORDS: Extrusion; potato snacks; GC-O; AEDA; SAFE

INTRODUCTION

Potato snacks occupy a great segment of the snack food market. The most popular are, beyond doubt, potato chips made either in the original way—cut from raw potatoes and fried—or via the process based on potato powder rehydration, extrusion, shaping, and finally deep frying (1). In each case the processing leads to a product with extremely high fat content (30–45%). In recent years, interest in low-fat and fatfree products has grown rapidly. It drives the snack food industry into finding new ways to obtain food products with lower oil content but retaining desirable texture and flavor. Extrusion is an important process in the production of such products. It is used worldwide to produce, among other, foods such as ready-to-eat breakfast cereals and snacks. During the extrusion process extremely high shear, high pressure, and temperature modify the physical and chemical properties of raw materials subjected to it.

In terms of flavor, the extrusion process causes chemical degradation due to oxidation, hydrolysis, and other reactions occurring inside the barrel as well as volatile component flush off. On the other hand, materials containing reducing sugars and free amino acids undergo the Maillard reaction expressed by color and flavor development (2). There is information on the sensory properties of extruded cereals from corn flour (3–

5), wheat (6), or oat flour (7), but only a few papers on the sensory properties of extruded potato products have been published. One of them is the work of Faller et al. (8), who investigated the effect of potato granule type, feed moisture, and addition of oil on the sensory attributes of extruded potato puffs. However, it is hard to find any information about the effect of raw material components and extrusion cooking processing conditions on the formation of characteristic volatiles corresponding to the overall flavor of extruded potato snacks.

Recent investigations on key odorants of boiled potatoes (9–11), baked potatoes (12, 13), or French fries (14) identified more than 150 volatile components, but only several of them contribute significantly to the aroma of these products. In those publications the most often mentioned compounds are methional, having a boiled potato flavor; 4,5-epoxy-(*E*)-2-decenal, metallic; dimethyl trisulfide, cabbage odor; several pyrazines, roasty or earthy; vanillin, vanilla-like; sotolon, spicy; furaneol, caramel-like; and 2-acetyl-1-pyrroline, roasty and popcorn-like (11). Compounds such as 1-octen-3-ol, (*Z*)-2-nonenal, (*E*)-2-nonenal, and (*E,E*)-2,4-decadienal, which are products of the oxidation of linoleic acid, and (*Z*)-1,5-octadien-3-one and (*E,Z*)-2,6-nonadienal, formed by the oxidation of linolenic acid (10, 15, 16), give the metallic, green, fatty flavor, which in higher concentration can resemble the flavor of boiled potatoes (9).

The aim of this study was to (i) identify potent odorants of potato snacks with low ($\leq 9\%$) fat content, obtained by extrusion

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cooking, and (ii) characterize flavor compounds in each processing stage of snack production. Flavor compounds were identified by gas chromatography–olfactometry (GC-O) and gas chromatography–mass spectrometry (GC-MS). To avoid the formation of artifacts during the isolation of volatiles, the solvent-assisted flavor evaporation (SAFE) method was applied for their extraction.

MATERIALS AND METHODS

Chemicals. Diethyl ether, *n*-pentane, 2,3-butanedione, butanal, heptanal, dimethyl trisulfide, 1-octen-3-ol, limonene, 2-acetylpyrazine, 2-ethyl-3,5-dimethylpyrazine, benzenemethanethiol, 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone, 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone, (*E,E*)-2,6-nonadienal, and 5-methyl-2,3-diethylpyrazine were obtained from Aldrich (Poznań, Poland), and hexanal, (*Z*)-2-hexenal, methional, and phenylacetaldehyde were from Fluka (Poznań, Poland). 2-Acetyl-1-pyrroline was obtained as a kind gift from Prof. P. Schieberle.

Preparation of Extruded Potato Snacks. For extruded potato snack production potato granules, wheat flour, canola oil, maltodextrin (DE-18), salt, and baking powder were used. Potato granules (8% moisture content) were obtained from Potato Processing Plant SOLAN S.A. located in Glowno, Poland. Canola oil, wheat flour (7% moisture content), salt, and baking powder were purchased in a local store, and maltodextrin was obtained from the Potato Research Laboratory, Poznań, Poland.

To find the optimum dough recipe several variations were verified: addition of oil, 0, 5, 7, and 9% (w/w); addition of salt, 0 and 1% (w/w); addition of baking powder, 0, 1, and 2% (w/w); addition of maltodextrin, 0, 5, and 10% (w/w); addition of wheat flour, 0, 15, and 25% (w/w); and water addition, 15, 20, 27, and 30% (w/w). On the basis of preliminary results and sensory analyses of products, the optimum recipe was developed: base potato snacks were prepared from potato granules (80%), wheat flour (15%), and maltodextrin (5%). To the base were added canola oil (7%), salt and baking powder (1% each), and water (27%). Potato granules and other ingredients were carefully mixed into a homogeneous material and then processed in a Werner Pflleiderer ZK 25 twin-screw cooking extruder. Optimum extrusion conditions were developed: temperatures of each of four baking sections were 25, 115, 115, and 90 °C, respectively. Screw speed was set to 50 rpm. After extrusion, products (still moist and flexible) were cut into small pieces and dried in an oven at 85 °C for 15 min followed by 45 min of heating at 130 °C to develop proper texture and aroma.

Sensory Analysis. Sensory analyses were evaluated by a 10 member sensory panel in a five-category ranking test using a scale from 0 to 5 points. The panelists determined odor, color, texture, flavor, and overall acceptance, giving notes from 0 for the poorest judged sample to 5 for the best one. Each extrudate was assessed three times by each judge, and average note was calculated for each attribute.

Isolation of Volatiles. Extruded and dried potato snacks (20 g) were ground and mixed with water (150 mL). The volatile fraction was isolated by high-vacuum distillation using the SAFE technique (17). During this procedure, the temperature of the water bath was held at 40 °C, and pressure was reduced using an Edwards RV5 rotary vane pump. Distillate with flavor compounds was collected in a flask cooled with liquid nitrogen. After 30 min of distillation, the solution was thawed and extracted five times with 10 mL of a pentane/ether mixture (1:1), and after drying over anhydrous Na₂SO₄, the fraction was concentrated to ~200 μL under a delicate stream of nitrogen.

Gas Chromatography–Olfactometry. GC-O was performed on an HP 5890 chromatograph using the following capillary columns: SPB-5 (30 m × 0.53 mm × 1.5 μm, Supelco, Bellefonte, PA) and Supelcowax-10 (30 m × 0.25 mm × 0.25 μm). The chromatograph was equipped with a Y splitter dividing effluent between an olfactometry port with humidified air as a makeup gas and a flame ionization detector. The operating conditions were the following: initial oven temperature, 40 °C (1 min), then raised at 6 °C/min to 180 °C and at 20 °C/min to 280 °C for the SPB-5 column; 40 °C (2 min), then raised to 60 °C at 40 °C/min rate, held for 2 min isothermally, and then raised at 5 °C/min to 240 °C for the Supelcowax-10 column. For all peaks and flavor

notes occurring at a specific retention times, retention indices were calculated to compare results with those obtained by GC-MS and data in the literature. Retention indices were calculated for each compound using homologous series of C₅–C₂₄ *n*-alkanes (18).

Gas Chromatography–Mass Spectrometry. Compound identification was performed using a Thermoquest Trace 2000 gas chromatograph coupled to a Finnigan PolarisQ ion trap mass spectrometer. The chromatograph was equipped with a fused silica DB-5 (30 m × 0.25 mm × 0.25 μm) column and the same Supelcowax-10 column used in the GC-O experiments. Operating conditions for GC-MS were as follows: helium flow, 0.8 mL/min; oven conditions, same as for GC-O. Mass spectra were recorded in electron impact mode (70 eV) at a source temperature of 200 °C. A scan range of *m/z* 33–350 was employed. Identification of volatiles was performed in different ways depending on the availability of standard compounds: full identification comprising comparison of mass spectra, retention indices (RI), and odor notes on two columns of different polarities was performed when a standard of the investigated compound was available. In some cases the MS signal of the analyte was too weak to facilitate mass spectra comparison. In such cases RI and odor notes of compounds were compared to standard. In cases when standards were not available, tentative identification was performed on the basis of the comparison of the mass spectrum of a compound with a NIST 98 library match and comparison of retention indices with that available in the literature. Also, an odor characteristic for an analyzed compound was compared with literature data and used in tentative identification.

Aroma Extract Dilution Analysis (AEDA). The flavor dilution factor (FD) of each of the odorants was determined by an AEDA (19). The flavor extract (2 μL) was injected into a GC column. Odor-active regions were detected by GC-effluent sniffing (GC-O), and three panelists determined the description of the volatiles. The extract was then stepwise diluted by addition of pentane/ether (1:1) at a ratio of 1+1 v/v, and each of the dilutions was analyzed until no odor was perceivable at the sniffing port. Retention data of the compounds were expressed as retention indices (RI).

RESULTS AND DISCUSSION

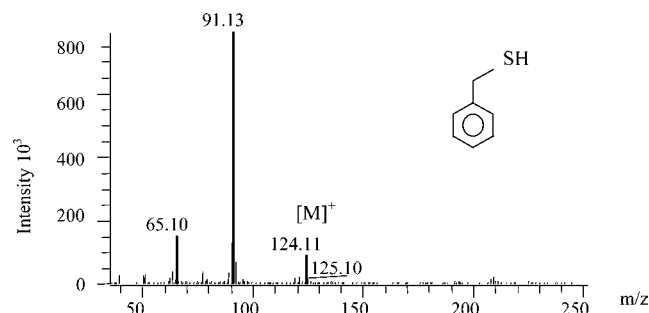
In preliminary experiments the extrusion cooking process (temperature of baking sections and screw speed) and drying conditions (temperature and time) were optimized to generate an attractive product with desirable texture and flavor. The dough containing 27% (w/w) of water, 7% (w/w) of canola oil, and salt and baking powder in amounts of 1% (w/w) of each was extruded under different temperatures (90, 115, 130, and 150 °C) and different screw speeds (20, 50, 90, and 120 rpm) and, after extrusion, dried in an oven with air recirculation at four temperatures (70, 85, 105, and 130 °C) for different times (0.5, 1, 1.5, and 3 h). Products were evaluated by a 10 member sensory panel in a five category ranking test using a scale from 0 to 5 points. As a result, the most attractive odor, color, texture, flavor, and overall acceptance were judged for a sample obtained under the following conditions: (i) temperatures of each baking section as 25, 115, 115, and 90 °C; (ii) screw speed of 50 rpm; (iii) drying parameters, 85 °C for 15 min and 130 °C for 45 min. This sample was used for flavor characterization. An aroma concentrate was then prepared from ground potato extrudates by SAFE and then extracted with a pentane/ether solution. Concentrated extract was analyzed for identification of odor active compounds by AEDA. To determine the origin of volatiles in snacks, volatiles from the dough prepared before extrusion, from the extrudate, and from the extrudate dried in an oven were analyzed.

In the analyzed extract prepared from the dough before extrusion processing, 13 compounds were identified showing FD factors in the range of 1–4096, but only 3 of them had high FD factors, which can contribute mostly to the flavor of potato granules (Table 1). Other compounds, although having

Table 1. Key Odorants Identified in Potato Granules Used for Extrusion of Potato Snacks (Isolation Performed Using SAFE Method)

no.	compound	odor description ^b	RI ^a		
			DB-5	Supelco-wax-10	FD ^c
1	unknown	fruity	698		1
2	hexanal ^d	green	801	1081	1
3	unknown	valerian	820	1112	2
4	<i>o</i> -xylene ^d	geranium	856	1183	64
5	heptanal ^d	rancid	893	1173	1
6	methional ^d	boiled potato	909	1456	128
7	2-acetyl-1-pyrroline ^d	popcorn	916	1322	2
8	dimethyl trisulfide ^d	cabbage	961	1377	1
9	1-octen-3-ol ^e	mushroom	982	1398	1
10	phenylacetaldehyde ^d	flowery	1053	1642	1
11	benzenemethanethiol ^d	garden cress seed	1135	1615	4096
12	unknown	parsley root	1185	1698	2
13	2,6-nonadienal ^d	cucumber	1190	1575	2

^a Retention index. ^b Odor description at sniffing port. ^c Flavor dilution factor determined on DB-5 type (SPB-5) column. ^d Compound identified by comparing its RI, mass spectrum, and odor descriptor with those of authentic standard. ^e MS signal too weak to obtain good spectrum. Identification based on comparison of odor description and RI with a standard compound. Identification based on comparison of mass spectrum and/or RI and odor notes with data in the literature.

**Figure 1.** Mass spectrum (EI) and a structure of benzenemethanethiol. Spectrum was obtained from a standard of this compound.

low FD factors, can also influence the overall flavor of analyzed product. The compound with the highest FD factor (4096) had a flavor of garden cress seed (*Lepidium sativum*). There has been no report on this type of flavor in any product, so necessary work was done to find out which compound was responsible for this odor. Garden cress seeds were grown on moist cotton and then collected and ground, and a Likens–Nikerson distillation/extraction was performed (20). The extract was analyzed by GC-O, and it was discovered that the compound corresponding to garden cress flavor is benzenemethanethiol, which was confirmed by comparing its EI mass spectrum with that of authentic standard and comparing its odor quality at the sniffing port. The mass spectrum of benzenemethanethiol and its structure are presented in **Figure 1**. The second most intense odor (FD 128) was described as boiled potato, which was represented by methional, and a third odor described as geranium was represented by *o*-xylene.

Fifteen volatiles were obtained from the extract prepared from extrusion product. They are shown in **Table 2**. More compounds had a significant FD factor that can contribute to the flavor of extruded potato granules. Once again the compound with the highest FD factor (4096) was benzenemethanethiol with garden cress seed odor. Other compounds with high FD factors were the following: two compounds with a flavor of boiled potatoes, an unknown one (FD 512) and methional (FD 16); 3-methylbutanal (FD 16) with malty flavor; dimethyl trisulfide (FD 16) with cabbage-like flavor; 1-octen-3-ol (FD 8), with mush-

Table 2. Key Odorants Identified in Potato Snacks after Extrusion and before Drying (Isolation Performed Using SAFE Method)^a

no.	compound	odor description ^b	RI ^a		
			DB-5	Supelco-wax-10	FD ^c
1	ethanol ^d	spirit	<500	<600	2
2	unknown	cumin	583	898	4
3	3-methylbutanal ^d	malty	646	901	16
4	hexanal ^d	green	801	1081	1
5	unknown	potato	866	1325	512
6	heptanal ^d	rancid	893	1173	2
7	methional ^d	boiled potato	909	1456	16
8	dimethyl trisulfide ^d	cabbage	961	1377	16
9	1-octen-3-ol ^e	mushroom	982	1398	8
10	1,5-octadien-3-one ^f	geranium	984	1358	1
11	unknown	sweet candy	1010	1534	1
12	phenylacetaldehyde ^d	flowery	1053	1642	2
13	benzenemethanethiol ^d	garden cress seed	1135	1615	1024
14	unknown	parsley root	1185	1698	4
15	2,6-nonadienal ^d	cucumber	1190	1575	1

^a Retention index. ^b Odor description at sniffing port. ^c Flavor dilution factor determined on DB-5 type (SPB-5) column. ^d Compound identified by comparing its RI, mass spectrum, and odor descriptor with those of authentic standard. ^e MS signal too weak to obtain good spectrum. Identification based on comparison of odor description and RI with a standard compound. ^f Tentative identification—no standard available. Identification based on comparison of mass spectrum and/or RI and odor notes with data in the literature.

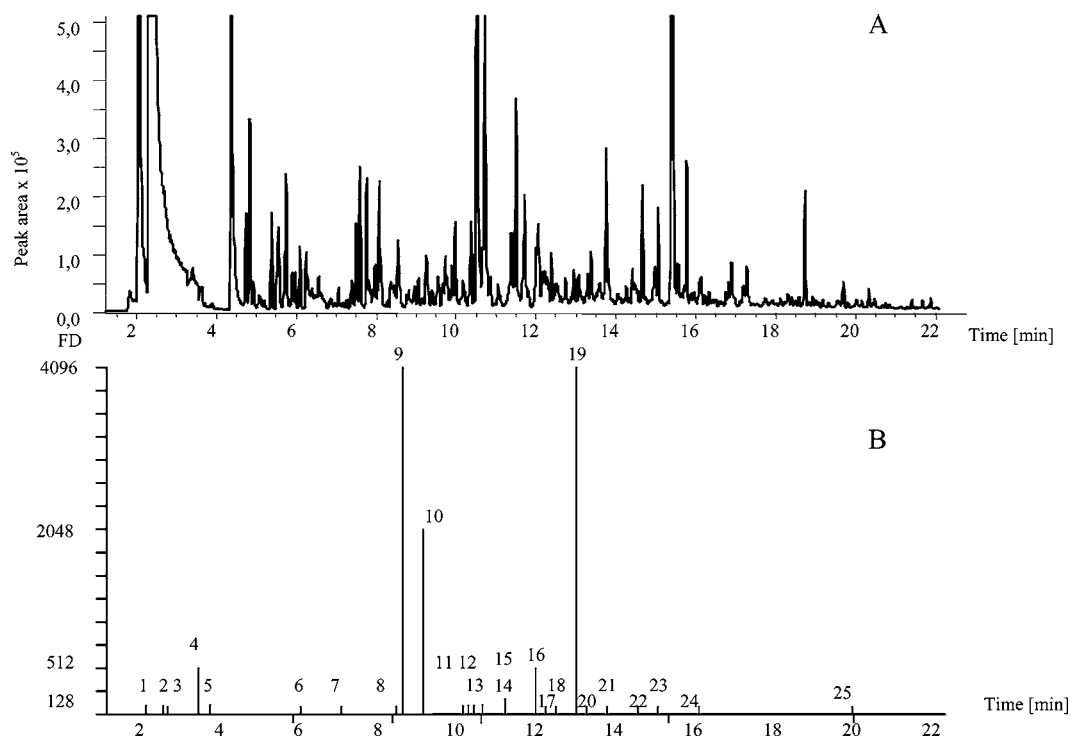
room-like flavor; and an unknown one (FD 4) with cumin-like flavor. From 15 compounds identified in extrudates 7 were not present in the aroma extract obtained from potato granules; therefore, they may be formed during the extrusion process. These compounds were ethanol, 3-methylbutanal, (*Z*)-1,5-octadien-3-one, and unknowns with the flavors of cumin, boiled potatoes, sweet candy, and parsley root. 3-Methylbutanal (FD 16) is generated by a Strecker degradation of leucine (21); it can be therefore assumed that it was formed in a similar way in the extruder barrel. On the other hand, (*Z*)-1,5-octadien-3-one belongs to the group of off-flavors, identified also in boiled potatoes (11) or French fries (14), generated in an oxidation pathway from linolenic acid.

GC-O performed for isolates obtained from extrudates dried in an oven showed 25 odor-active compounds having FD factors in the range of 2–4096 (**Table 3**). The total ion chromatogram and aromagram of this sample are shown in **Figure 2**. Among dried extrudate volatiles methional (FD 4096), benzenemethanethiol (FD 4096), 2-acetyl-1-pyrroline (FD 2048), phenylacetaldehyde (FD 512), butanal (FD 512), and 2-acetylpyrazine (FD 128) were considered to be the main contributors to the odor of extruded potato snacks. Methional, with a boiled potato flavor, has also been characterized as a key contributor to the flavor of boiled potatoes (11), wheat bread crust (22), roasted wild mango seeds (23), or popcorn (24). Benzenemethanethiol, which shows the highest FD factor in dough prepared for extrusion, in potato extrudates, and in dried potato extrudates, was not reported to be present in any other product. From our preliminary studies it also appears as a main contributor to the flavor of garden cress seed (*L. sativum*). 2-Acetyl-1-pyrroline has been previously identified in toasted bread (25), popcorn (26), aromatic rice (27), or roasted sesame seeds (28). Phenylacetaldehyde is a common component of products such as French fries (14), Camembert cheese (29), cooked brown rice (30), or wheat bread crust (22). 2-Acetylpyrazine is reported to be present in popcorn (26), French fries (14) or wheat bread crust (22).

Table 3. Key Odorants Identified in Extruded Potato Snacks after Drying (Isolation Performed Using SAFE Method)

no.	compound	odor description ^b	RI ^a		FD ^c
			DB-5	Supelco wax-10	
1	ethanol ^d	spirit	<500	<600	8
2	methanethiol ^e	burnt	500	695	8
3	2,3-butanedione ^d	buttery	593	1054	4
4	butanal ^d	rancid	596	831	512
5	unknown	fruity	683		32
6	hexanal ^d	green	801	1081	8
7	(Z)-2-hexenal ^d	fatty	855	1204	8
8	heptanal ^d	rancid	893	1173	32
9	methional ^d	boiled potato	909	1456	4096
10	2-acetyl-1-pyrroline ^d	popcorn	916	1322	2048
11	dimethyl trisulfide ^d	cabbage	961	1377	64
12	1-octen-3-ol ^e	mushroom	982	1398	64
13	1,5-octadien-3-one ^f	geranium	984	1358	64
14	limonene ^e	lemon	1015	1178	4
15	2-acetylpyrazine ^d	roasty	1023	1604	128
16	phenylacetaldehyde ^d	flowery	1053	1642	512
17	2-ethyl-3,5-dimethylpyrazine ^d	earthy	1081	1437	8
18	4-hydroxy-2,5-dimethyl-3(2H)-furanone ^e	cotton candy	1099	2030	32
19	benzenemethanethiol ^d	garden cress seed	1135	1615	4096
20	3-hydroxy-4,5-dimethyl-2(5H)-furanone ^f	spicy, seasoning-like	1178	2193	4
21	2-methyl-3,5-dimethylpyrazine ^f	parsley root	1185	1474	16
22	2,6-nonadienal ^d	cucumber	1190	1575	32
23	5-methyl-2,3-diethylpyrazine ^d	roasted pepper	1212	1483	8
24	unknown	fresh pepper	1248	1498	8
25	β -damascenone ^f	fruity, sweet	1386	1823	2

^a Retention index. ^b Odor description at sniffing port. ^c Flavor dilution factor determined on DB-5 type (SPB-5) column. ^d Compound identified by comparing its RI, mass spectrum, and odor descriptor with those of authentic standard. ^e MS signal too weak to obtain good spectrum. Identification based on comparison of odor description and RI with a standard compound. ^f Tentative identification—no standard available. Identification based on comparison of mass spectrum and/or RI and odor notes with data in the literature.

**Figure 2.** Total ion chromatogram (A) and aromagram (B) of volatile compounds of extruded and dried potato snacks. Compound numbers correspond to the numbers listed in Table 3.

Methional and phenylacetaldehyde are generated during Strecker degradation of the amino acids methionine and phenylalanine, respectively (21, 31). 2-Acetyl-1-pyrroline has been shown to be formed by an acylation of 1-pyrroline by the respective 2-oxoaldehyde with elimination of formaldehyde (26). Those compounds were found in extruded potato snacks, as well as in potato granules used for extrusion processing, so it could

be concluded that either they were developed during production of potato granules or they were already present in raw potato. It has been reported that methional and 2-acetyl-1-pyrroline are not present in raw potatoes (10, 31); however, phenylacetaldehyde has been found in both raw and boiled potatoes (32). Butanal (FD 512), with an unpleasant rancid flavor, was probably formed via oxidation by degradation from either

linoleic or linolenic acid, as well as hexanal, (*Z*)-2-hexenal, heptanal, (*Z*)-1,5-octadien-3-one, and (*E,E*)-2,6-nonadienal. All of these compounds had FD factors of >8.

From 25 compounds identified in extruded and dried potato snacks methanethiol, 2,3-pentanedione, limonene, 2-acetylpyrazine, 2-ethyl-3,5-dimethylpyrazine, 4-hydroxy-2,5-dimethyl-3(*2H*)-furanone, 3-hydroxy-4,5-dimethyl-2(*5H*)-furanone, 2-methyl-3,5-diethylpyrazine, 5-methyl-2,3-diethylpyrazine, and β -damascenone were not found either in a dough prepared for extrusion or in extruded potatoes, so it could be concluded that they were formed during the drying process. Methanethiol is a compound that is formed during the heating of food from methionine by Strecker degradation (21). 2-Ethyl-3,5-dimethylpyrazine, 5-methyl-2,3-diethylpyrazine, and acetylpyrazine are often reported in processed food products such as popcorn (24), rye bread crust (22), or roasted wild mango fruits (23) because of their very low odor threshold values: 0.04 $\mu\text{g/L}$, 0.09 $\mu\text{g/L}$, and 0.4 ng/L, respectively (21, 22). Pyrazines 17 and 23 in Table 3 are formed from 2-oxopropanal and alanine by Maillard reaction. Compounds such as furaoneol [4-hydroxy-2,5-dimethyl-3(*2H*)-furanone] and sotolon [3-hydroxy-4,5-dimethyl-2(*5H*)-furanone] were also formed in the course of the Maillard reaction, for example, furaoneol from 2-hydroxypropanal and its oxidation product, 2-oxopropanal, and sotolon from 2,3-butanedione and glycolaldehyde (21). β -damascenone, which is present in tomato (33), black tea (34), and raw arabica coffee (35), is reported to be formed in the oxidative degradation of carotenoids.

Drying of potato snacks after extrusion was a crucial technological step in their production, yielding both acceptable physical properties (texture and crunchiness), golden color, and full flavor. Although benzenemethanethiol was a crucial flavor impact compound, having the highest FD value, in granulate, extrudate, and dried snacks, only snacks after drying achieved a highly desirable flavor. Apart from benzenemethanethiol, flavor was influenced to the highest extent by methional, 2-acetyl-1-pyrroline, butanal, and phenylacetaldehyde. The flavor of dried snacks was more pronounced—21 of 25 compounds had a FD value of >8 compared to 6 of 15 in snacks before drying. However, the full answer on the impact of particular compounds on potato snack flavor can be gained only by estimating odor-active values for all of them. It was possible to obtain low-fat snacks based on potato granules with attractive flavor. Further research on influencing the flavor profile of this product by the addition of aroma precursors is being conducted.

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