Determination of quality characteristics for whole milk powder with slight and moderate odour changes

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Summary

Whole milk powder is susceptible to flavour changes, which is limiting for its shelf-life as well as consumer acceptability. Degradation of natural aroma during production and storage occurs primarily as a result of lipid oxidation, Maillard reactions and the influence of extracellular microbial enzymes. Selected chemical parameters were measured in ten whole milk powder samples to assess their susceptibility to off-odour development. The results of off-odour intensity were correlated with the volatile compounds determined by headspace solid-phase microextraction (HS-SPME) coupled to gas chromatography-mass spectrometry (GC-MS) and with other physicochemical parameters (moisture, protein content, content of organic acids and their salts, peroxide value and content of free fat). The off-odour intensity correlated with contents of volatile compounds, lactic acid, proteins, propanoic acid, moisture and peroxide value. Based on the results, characteristics of a moderate off-odour intensity, which is still acceptable to consumers, were established to predict flavour changes with the following limit values: total content of volatiles with the sum of peak areas of max. 400 V·s, content of hexanal of max. 10 $\mu g \cdot kg^{-1}$, content of lactic acid of max. 1 $g \cdot kg^{-1}$, protein content of min. 250 $g \cdot kg^{-1}$, moisture of max. 30 $g \cdot kg^{-1}$, content of propanoic acid of max. 1 $g \cdot kg^{-1}$ and peroxide value of max. 5 milliequivalents O_2 per kilogram of fat.

Keywords:

milk powder; odour changes; volatile components; sensory properties

Flavour stability of whole milk powder (WMP) is one of the most important aspects limiting its shelf-life as well as consumer acceptability of the reconstituted milk or products made from WMP. The flavour character depends on sensory active compounds, typical sensory characteristics of fresh WMP being a slightly sweet taste, a mild and pleasant flavour with a slightly cooked aroma but free from off-flavours and taints [1].

WMP is susceptible to aroma-active compounds formation and, consequently, to changes of sensory properties during its production and storage [1, 2]. Whereas some level of these changes is natural and may improve the characteristic aroma, their increased intensity can cause undesirable sensory defects. Sensory quality of WMP depends on the milk composition, which can be primarily influenced by genetic predispositions of cows, microbiological quality of fluid milk, feed composition, and the season or geographical conditions. Moreover, process conditions, such as temperature and time of drying, packaging and storage conditions have been deemed the most critical factors affecting WMP flavour [2–4]. Some physicochemical parameters, such as heating temperature, pH value, water activity or moisture, are adjusted during WMP production to achieve a proper balance of biochemical changes, which increases the quality of the final product.

Whetstine et al. [1] identified more than 60 aroma-active compounds in fresh and stored WMP. These compounds originate as a result of a combination of physico-chemical and enzymatic reactions of the main milk components, i.e. lactose, milk fat and proteins. The most important compounds causing off-flavours are produced by lipid oxidation (particularly hexanal and other straight-chain aldehydes and ketones), protein

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degradation (Stecker aldehydes, methional, dimethyl disulphide and others), and reactions of extracellular microbial enzymes (short-chain free fatty acids) or by absorption of aromatic substances from the surroundings [1, 2, 5]. Headspace solid-phase microextraction (HS-SPME) is a convenient method for extraction of volatile compounds in WMP and gas chromatography-mass spectrometry (GC-MS) for their separation and detection [2, 3, 6]. However, determination of the cause of undesirable changes of WMP flavour is complicated by the fact that many aroma-active compounds are produced by two or more mechanisms [1, 2, 4]. Therefore, it is essential to monitor a set of attributes.

The aim of the study was to assess susceptibility of a set of WMP samples to undesirable odour changes on the basis of determination of relevant chemical parameters, and consequently to determine characteristics of a moderate off-odour intensity. Off-odour intensity of the samples was assessed using sensory evaluation and the results were correlated with the chromatographic measurement of volatile compounds and with physico-chemical parameters.

MATERIALS AND METHODS

Materials

Ten instant WMP samples (designated S1–S10) and one control sample (designated SC) were collected from a local production facility in Czech Republic. The samples (S1–S10) were analysed four to six months after their production (Tab. 1). Until analysis, they were stored in their original re-closable plastic packages under the recommended storage conditions (i.e. at 20 °C and in a dark place). The control sample was fresh, i.e. less than one month old. The samples contained 280 $g \cdot kg^{-1}$ of fat and were manufactured by spray drying of pasteurized full-fat cows' milk. Lecithin was used in the final part of their production to assure sufficient dissolution in water. Due to a relatively low variability among the samples in the dry matter content, all results of the analyses were expressed on fresh weight basis. The standard compounds hexanal (≥98.0%), *n*-butyl acetate (analytical standard), potassium hydrogen phthalate (≥99.9%), propanoic acid (≥99.5%) and DL-lactic acid lithium salt (98.0%) were purchased from Sigma Aldrich (St. Louis, Missouri, USA); sodium acetate trihydrate (≥99.0%) was purchased from Lach-Ner (Neratovice, Czech Republic).

Sensory evaluation

For the sensory analysis, a difference-from-control test was used [7]. The off-odour difference between the control sample (SC) and the ten test samples (S1–S10) was evaluated by a team of 20 panellists (from among the permanent staff of Department of Food Preservation, University of Chemical Technology Prague, Czech Republic), who were trained and monitored according to the relevant international standards [8, 9]. All WMP samples were reconstituted to a 100 $g \cdot l^{-1}$ solution with odour-free distilled water at laboratory temperature [2]. The panellists rated the samples by giving marks on a numerical category scale, where 0 was "no difference from the control" and 9 was "very large difference from the control" (each of the test samples was evaluated once). Five samples were assessed per a single session. The mean rates were compared with the control by Tukey's test (at $p < 0.05$). Off-odour intensity was evaluated in the control sample. The panellists scored the off-odour intensity on an intensity scale ranging from 0% to 100% between the two extremes: "off-odour free" and "strong off-odour, unacceptable for consumers".

Basic quality parameters

Moisture was determined by drying to constant weight in an oven at a temperature of 102 °C [10]. Organic acids and their salts (lactic acid, acetic acid and propanoic acid) were determined by capillary isotachophoresis with conductivity detection [11]. Determination of protein content was based on quantitative determination of amino acids using formaldehyde titration of a known amount of reconstituted milk with 0.25 $mol \cdot l^{-1}$ sodium hydroxide [12]. Peroxide value was determined using potentiometric titration according to modified AOAC method, with extraction of lipids being performed by a cold mixture of chloroform and methanol (2:1, v/v) [13]. After filtration, an aliquot was dissolved in a mixture of chloroform and acetic acid (2:3, v/v) and titration using 0.002 $mol \cdot l^{-1}$ sodium thiosulfate was carried out. Free fat was determined by extraction with petroleum ether and it refers to the percentage of extracted substances [14].

Headspace analysis of volatiles

Volatiles were analysed using HS-SPME/GC-MS according to LLOYD et al. [2] and BIOLATTO et al. [3] with modifications. Divinylbenzene/carboxen/polydimethylsiloxane fibre (50/30 μm; Supelco, Bellefonte, Pennsylvania, USA) was inserted into a 10 ml headspace vial filled with 500 mg of a sample, 2 ml of odour-free deionized

water, and 10 μl of internal standard (10 mg of *n*-butyl acetate per litre of ethanol), and agitated at 8.33 Hz (used for hexanal quantification). The sample tempering took 1 min at 40 °C, the sample extraction took 10 min at 40 °C and desorption took 4 min at 240 °C.

The gas chromatography conditions were as follows: the sample analyses were performed using a gas chromatograph (7890A) equipped with a mass spectrometry detector (5975C) and the HP-5MS 5% diphenyl 95% dimethylpolysiloxane column (30 m × 0.25 μm × 0.25 μm), all from Agilent Technologies (Santa Clara, California, USA). The temperature programme was as follows: the initial temperature of 60 °C was held for 2 min, then ramped to 320 °C at 10 °C·min^{-1} and held at 320 °C for 2 min. The detector temperature was as follows: 230°C mass spectrometry (MS) source and 150 °C MS Quadrupole. The carrier gas (helium) flow was 1.4 ml·min^{-1}. Volatile compounds were identified using NIST 08 MS Database (National Institute of Standards and Technology, Gaithersburg, Maryland, USA), retention indeces [1, 2] and by comparison with mass spectra of standard compounds injected under the same conditions. With regard to that approach to evaluation, the method was verified only in terms of repeatability for 5 major volatile aldehydes; repeatability expressed as relative standard deviation (*RSD*) was less than 10.0% for each of them.

Statistical analysis

The tests of basic quality parameters were done in triplicate, analyses of volatiles in duplicate for each sample, and the mean values are reported. Data were statistically analysed by one-way analysis of variance (ANOVA) to determine significant differences (*p*-value) among samples and averages were compared by Tukey's test. The results of sensory evaluation were correlated with the results for quality parameters and volatiles, to evaluate the effect that various parameters have on off-odour development. Furthermore, principal component analysis (PCA) was used to find the relationships among samples. These statistical analyses were performed using Excel 2007 (Microsoft, Redmond, Washington, USA) and Statistica CZ 12 (Stat Soft, Prague, Czech Republic).

RESULTS AND DISCUSSION

Sensory evaluation

The average value of the off-odour inten-

Tab. 1. Sensory evaluation, basic quality parameters and volatiles of whole milk powders.

	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
Time from production [week]	25	24	23	22	21	20	19	18	17	16
Off-odour difference	5.3 ± 1.6^{ab}	4.3 ± 1.4^{bc}	5.5 ± 5.5^{ab}	5.6 ± 1.4^{a}	3.2 ± 0.9^{c}	1.3 ± 1.0^{d}	1.5 ± 1.2^{d}	1.8 ± 1.4^{d}	1.7 ± 1.1^{d}	1.4 ± 1.1^{d}
Moisture [g·kg^{-1}]	25.17 ± 0.23^{c}	24.52 ± 0.57^{c}	32.04 ± 0.30^{b}	38.80 ± 1.49^{a}	25.27 ± 0.96^{c}	19.01 ± 0.18^{d}	34.12 ± 2.13^{b*}	16.45 ± 0.35^{de}	12.93 ± 0.20^{f}	14.62 ± 0.17^{ef}
Proteins [g·kg^{-1}]	237.64 ± 5.58^{de}	254.27 ± 1.52^{bc}	242.60 ± 5.32^{cd}	236.81 ± 3.18^{de}	226.63 ± 4.37^{e}	260.69 ± 0.53^{b}	255.92 ± 0.18^{b*}	256.01 ± 0.53^{b}	263.82 ± 6.33^{b}	280.35 ± 2.76^{a}
Peroxide value [meq·kg^{-1}]	1.48 ± 0.02^{c}	1.53 ± 0.01^{c}	6.14 ± 0.13^{b}	12.37 ± 0.59^{a}	1.12 ± 0.05^{c}	1.62 ± 0.22^{c*}	1.56 ± 0.16^{c}	1.62 ± 0.15^{c}	1.29 ± 0.04^{c}	0.57 ± 0.06^{c}
Propanoic acid [g·kg^{-1}]	1.26 ± 0.03^{a}	1.29 ± 0.05^{a}	1.24 ± 0.05^{a}	nd	nd	nd	nd	nd	nd	nd
Lactic acid [g·kg^{-1}]	1.13 ± 0.04^{c}	1.74 ± 0.07^{a}	1.34 ± 0.04^{b}	1.25 ± 0.02^{bc}	0.24 ± 0.03^{ef}	0.39 ± 0.04^{de}	0.46 ± 0.04^{d*}	0.11 ± 0.01^{f}	0.43 ± 0.01^{f}	0.37 ± 0.04^{de}
Acetic acid [g·kg^{-1}]	nd	0.70 ± 0.06^{a}	0.79 ± 0.07^{a}	nd	0.22 ± 0.02^{b}	0.64 ± 0.04^{a*}	0.16 ± 0.02^{b}	nd	nd	nd
Free fat [g·kg^{-1}]	17.63 ± 0.50^{b}	19.07 ± 0.04^{b}	81.15 ± 1.71^{a}	13.80 ± 0.19^{cd}	12.69 ± 0.30^{d}	15.04 ± 0.05^{c}	19.05 ± 0.61^{b*}	17.51 ± 0.10^{b}	13.68 ± 0.10^{cd}	17.73 ± 0.19^{b}
Volatile compounds [V·s]	710.9 ± 5.6^{b}	490.8 ± 21.1^{c}	3 391.4 ± 79.0^{a}	3 328.9 ± 3.8^{a}	125.4 ± 10.1^{de}	72.0 ± 0.8^{e}	104.5 ± 6.2^{de}	106.9 ± 4.2^{de}	233.5 ± 17.9^{d*}	195.9 ± 20.8^{de}
Hexanal [μg·kg^{-1}]	28.45 ± 0.18^{b}	17.97 ± 1.22^{c}	187.77 ± 3.83^{a}	181.05 ± 1.58^{a}	2.14 ± 0.03^{d}	1.97 ± 0.07^{d}	4.02 ± 0.33^{d}	3.87 ± 0.11^{d}	10.04* ± 0.61^{d}	8.48 ± 0.05^{d}

Results are expressed as average value ± standard deviation. The values with different small letters within a row are different ($p < 0.05$). The off-odour intensity of the control sample was 9.5% ± 2.9%. Peroxide values are expressed as milliequivalents O_2 per kilogram of fat. Volatile compounds are expressed as sum of peak areas.
* – limiting values of parameters for the samples whose off-odour intensity was not significantly different from the control sample (i.e. samples S6–S10), nd – not detected.

sity of the control sample was 9.5% and it can be described as very slight. The off-odour intensity of the test samples was evaluated by comparison with the control sample. Each of the test samples (S1–S10) was assessed 20 times against the control sample to determine the degree of difference. The off-odour intensity with the off-odour difference ranging between 1 and 3 could be characterized as slight, between 4 and 6 as moderate (still acceptable for consumers), and between 7 and 9 as strong (unacceptable for consumers). The results of the difference-from-control test (Tab. 1) indicated that the off-odour intensity of the control sample was not significantly different ($p < 0.05$) from samples S6–S10 (the maximum value of the off-odour difference in this sample group was 1.75). The off-odour difference of samples S1–S5 from the control sample ranged between 3.15 and 5.60.

Basic characterization of the samples

A set of quality parameters was analysed in relation to potential undesirable off-odour formation. The average values and the results of the analysis of variance are presented in Tab. 1.

The results of moisture ranged from 12.93 g·kg^{-1} to 38.80 g·kg^{-1}. According to Lloyd et al. [2], the optimal value of moisture of WMP should be below 30.0 g·kg^{-1} to ensure flavour stability; it was indicated that Maillard reactions may occur at moisture above 50.0 g·kg^{-1}. Celestino et al. [15] mentioned that changes in moisture during storage can be caused by changes in the state of lactose since lactose tends to absorb moisture from environment and shifts from amorphous to α-crystalline form.

According to Lloyd et al. [16], WMP contains between 245.0 g·kg^{-1} and 270.0 g·kg^{-1} of proteins; our results ranged between 226.63 g·kg^{-1} and 280.35 g·kg^{-1}. To ensure good solubility during WMP reconstitution, it is necessary to maintain the initial dispersion of the proteins when drying the milk. Celestino et al. [17] claimed that the protein content of milk powder can decrease during storage because of non-enzymatic browning.

The peroxide value ranged from 0.57 meq·kg^{-1} to 12.37 meq·kg^{-1}. Peroxide value corresponds to the content of hydroperoxides, the primary oxidation products, which indicates the initial phase of oxidation. Although hydroperoxides are not sensorially active, they are unstable and in the presence of oxygen can be decomposed through various mechanisms into low molecular weight compounds causing off-flavours already at very low contents [1, 18]. An increase in peroxide value can be expected during storage at temperatures above 30.0 °C, as a result of light exposure, inappropriate packaging in the presence of transition metals or in WMP fortified with fat with a higher content of polyunsaturated fatty acids [5, 18]. WMP shelf-life can be, however, prolonged by preventive measures, such as nitrogen-flushed packaging [18], antioxidant addition and pre-heating of the milk

Tab. 2. Major volatile compounds of whole milk powder samples and their flavour characteristics.

Compound	Retention time t_R [min]	Peak areas [V·s]			Average relative representation [%]	Characteristic flavour according to literature	References
		Range	Median	Mean			
Pentanal	1.9	13–196	60	71	8.1	Rubber	[21]
2-Hexanone	2.7	nd–15	0	3	0.3	Cooked	[4]
Hexanal	2.8	40–1 812	102	445	50.7	Green, grass, oxidized	[1, 2, 4, 21]
2-Heptanone	4.0	nd–458	14	74	8.4	Soapy, spicy, heat abuse, cooked	[2, 4]
Heptanal	4.1	9–571	18	139	15.8	Fatty, chemical, burnt, oxidized	[2, 4, 21]
Octanal	5.8	nd–216	7	51	5.8	Citrus, green, fragrant, citrus, oxidized (cardboard)	[1, 2, 4, 21]
3-Octen-2-one	6.4	nd–108	0	13	1.5	Fatty, mushroom	[2]
2-Nonanone	7.2	nd–47	0	9	1.0	Fresh, sweet, heat abuse, cooked	[4, 21]
(*E, Z*)-3,5-Octadien-2-one	7.2	nd–117	0	13	1.5	Synthetic, plastic	[21]
Nonanal	7.4	nd–188	16	59	6.7	Fatty, citrus, oxidized, cooked	[1, 2]

t_R – retention time, nd – not detected.

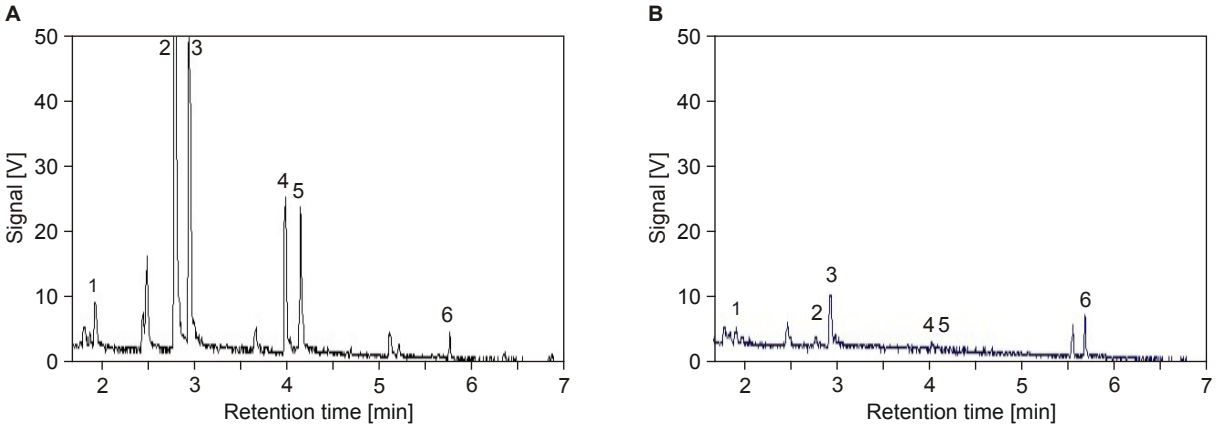

Fig. 1. Chromatograms of volatile compounds of whole milk powder.

A – sample of a moderate off-odour intensity (S3), B – sample of a slight off-odour intensity (S10).
The most significant differences were recorded in case of: 1 – pentanal (with retention time t_R = 1.9 min), 2 – 2-hexanone (t_R = 2.7 min), 3 – hexanal (t_R = 2.8 min), 4 – 2-heptanone (t_R = 4.0 min), 5 – heptanal (t_R = 4.1 min) and 6 – octanal (t_R = 5.8 min).

(at least 95 °C for 20 s), which leads to liberation of sulfhydryl groups with antioxidant effects, from the proteins [19].

Another analysed parameter was content of mineral substances, which are found in milk in the form of inorganic ions and salts, or as a part of organic compounds, namely, proteins, fats, carbohydrates and nucleic acids. Lactose and citrate are substrates of lactic fermentation by lactic acid bacteria [20]. In our study, the three major organic acids and their salts (lactic acid, acetic acid and propanoic acid) were determined by capillary isotachophoresis. The highest content was recorded for lactic acid ranging between 0.11 g·kg^{-1} and 1.74 g·kg^{-1}. Whetstine et al. [1] stated that the content of acetic acid (responsible for vinegar odour) and propanoic acid (responsible for Swiss cheese odour) in WMP decreased during storage. In our study, propanoic acid was detected only in samples with the off-odour intensity significantly different from the control sample (i.e. samples S1–S3).

Free fat contents ranged between 12.69 g·kg^{-1} and 81.15 g·kg^{-1}. According to Lloyd et al. [16], free fat usually ranges between 20.0 g·kg^{-1} and 33.0 g·kg^{-1}. Celestino et al. [15] stated that a decrease in free fat can be due to hydrolysis during storage, which is attributed to lipase activity.

Volatile components

Ten major volatile compounds were identified in the samples, including straight-chain aldehydes (pentanal, hexanal, heptanal, octanal and nonanal) and ketones (2-hexanone, 2-heptanone, 3-octen-2-one, 2-nonanone and 3,5-octadien-2-one). These compounds naturally occur in WMP in low contents; higher content indicates oxidative changes and can be responsible for non-standard flavours [1–3]. Their total contents in the analysed samples were highly variable: the total amount of volatiles expressed as sum of peak areas ranged from 72 V·s to 3391 V·s. The results of HS-SPME/GC-MS and reported characteristics of off-odours associated with the volatiles are summarized in Tab. 2. The chromatograms (Fig. 1) obtained from the sample of a slight off-odour intensity (sample S10) and the sample of a moderate off-odour intensity (sample S3) illustrate different profiles of the volatiles. Most of the identified compounds are formed by oxidation of unsaturated fatty acids, e.g. hexanal and 3-octen-2-one are produced from linoleic acid, whereas octanal and nonanal by oxidation of oleic acid [1, 2]. Moreover, unsaturated fatty acids are precursors of other aromatic compounds, such as methyl ketones, alcohols, lactones and esters [1].

Hexanal was found to be the most abundant volatile in all samples; its area ranged between 40 V·s and 1812 V·s and its content was from 2.02 μg·kg^{-1} to 194 μg·kg^{-1}. It was reported that content of hexanal in freshly produced WMP was less than 10 μg·kg^{-1} and, after having been stored for more than 4 months at 30 °C, exceeded 100 μg·kg^{-1}, while its flavour threshold in whole milk is about 50 μg·kg^{-1} [2, 22].

Relations among the parameters

The results of off-odour intensity determined as the difference from the control standard were correlated with quality parameters (Tab. 3). From the results, it was evident that off-odour difference

Tab. 3. Correlation between the results of off-odour difference and the results of quality parameters and volatiles of whole milk powders.

	Off-odour difference	Moisture	Protein content	Peroxide value	Propanoic acid	Lactic acid	Acetic acid	Free fat	Volatile compounds	Hexanal
Off-odour difference	1.00									
Moisture	0.65 [a]	1.00								
Protein content	–0.70 [a]	–0.63 [a]	1.00							
Peroxide value	0.64 [a]	0.71 [a]	–0.42	1.00						
Propanoic acid	0.70 [a]	0.23	–0.29	0.02	1.00					
Lactic acid	0.83 [a]	0.20	–0.13	–0.05	0.95 [a]	1.00				
Acetic acid	0.22	0.18	–0.08	0.01	0.52	0.64 [a]	1.00			
Free fat	0.43	0.31	–0.15	0.27	0.54	0.49	0.59 [a]	1.00		
Volatile compounds	0.77 [a]	0.68 [a]	–0.43	0.90 [a]	0.33	0.25	0.23	0.64 [a]	1.00	
Hexanal	0.75 [a]	0.67 [a]	–0.41	0.90 [a]	0.30	0.59 [a]	0.23	0.65 [a]	1.00 [a]	1.00

a – correlation significant on a level $\alpha = 0.05$ ($n = 10$).

strongly correlated with contents of volatile compounds ($r = 0.77$), in particular hexanal ($r = 0.75$). This agrees with the literature [2], where hexanal, 2-heptanone, and nonanal were determined as the predictors of a grassy flavour, and hexanal, octanal and 3-octen-2-one as the predictors of a painty flavour. Other important parameters influencing off-odour formation included lactic acid ($r = 0.83$), protein content ($r = –0.70$), propanoic acid ($r = 0.70$), moisture ($r = 0.65$) and peroxide value ($r = 0.64$). Correlation between free fat and off-odour difference was not confirmed ($r = 0.43$), which is consistent with Lloyd et al. [2]. The results on the correlation of off-odour intensity and volatiles, peroxide value and free fat confirmed the findings of a previous study, in which 20 samples of different production dates were tested [23].

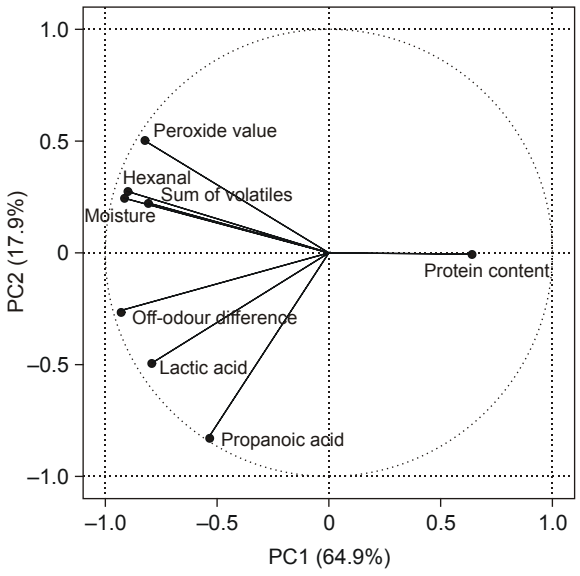

Fig. 2. Principal component plot of loadings for the tested whole milk powder samples.

Each point represents a triplicate or a duplicate analysis from ten samples (only parameters with the correlation coefficient higher than the critical value were included).

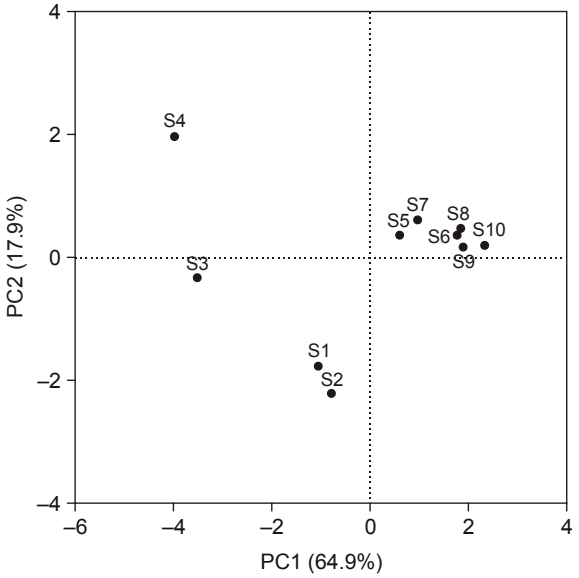

Fig. 3. Principal component plot of scores for whole milk powder samples.

Only the results of the parameters with the correlation coefficient higher than the critical value were included.

Off-odour development is a complex phenomenon, which depends on many parameters. Simple linear correlation focuses only on relationships between two selected variables. Since direct linear correlation was not so demonstrative due to the complexity of the reactions causing flavour changes in WMP, PCA was applied to reveal deeper structures in the data set. The original variables were linearly transformed into uncorrelated principal components, which represent a linear combination of the original variables. Fig. 2 presents the plot of loadings obtained for the first two principal components of the tested WMP sample set. Two principal components accounted for 82.8% of the total variance. Total amount of volatiles, hexanal content, moisture and lactic acid content were the descriptors with the highest contribution to off-odour intensity. These are on the right (positive) side of PC1. Scores for all WMP samples are shown in Fig. 3. Three clusters are evident; the first one (samples S5–S10) appears on the right (positive) side of PC1, while the second one (samples S1 and S2) and the third one (samples S3 and S4) are on the left (negative) side. The samples on the left side have the longest storage time; their off-odour intensity was evaluated as moderate; their contents of volatile compounds and hexanal, respectively, were significantly higher (in particular samples S3 and S4). The off-odour intensity of the most samples on the right side (samples S6–S10) was not significantly different from the control sample and it was evaluated as slight.

CONCLUSIONS

It is necessary to monitor a set of chemical parameters to assess susceptibility of whole milk powder to odour changes due to complexity and diversity of potential changes. It has been found that off-odour intensity significantly correlates with contents of volatile compounds, the most abundant volatile in all the samples being hexanal [2, 3]. We confirmed the correlation of off-odour intensity with moisture and peroxide value, our results correspond to the literature data [1, 2, 18]. Determination of other parameters, such as contents of proteins, lactic acid and propanoic acid, is also convenient for better prediction of odour changes [1, 16]. We suggested critical values for the mentioned parameters on the basis of our results obtained for samples whose off-odour intensity was not significantly different from the control sample. The following limits could serve as a tool for predicting undesirable odour changes: total amount of volatiles expressed as sum of peak areas (max. 400 V·s), hexanal content (max. 10 $\mu g \cdot kg^{-1}$), lactic acid content (max. 1 $g \cdot kg^{-1}$), protein content (min. 250 $g \cdot kg^{-1}$), moisture (max. 30 $g \cdot kg^{-1}$), propanoic acid content (max. 1 $g \cdot kg^{-1}$) and peroxide value (max. 5 milliequivalents O_2 per kilogram of fat). The more parameters do not keep to the mentioned limits, the greater is the susceptibility to off-odour development. All our results on the parameters of samples S6–S10, whose off-odour intensity was evaluated as slight, were within the limits; samples S2 and S5 exceeded the limits in one or two of the parameters and samples S1, S3 and S4 exceeded more than three of the parameter limits.

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