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ORIGINAL PAPER

Aroma stability and sensory aspects of commercially produced orange juice: gas chromatography–olfactometry study

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Abstract
The effects of pasteurization and 4-month storage on the aroma profile of orange juices with pulp that were fully processed and packed in inert nitrogen atmosphere were investigated within the span of 2 years (2018 and 2019). Headspace solid-phase microextraction, gas chromatography-mass spectrometry, as well as gas chromatography coupled to flame ionization detection and olfactometry, were used for extraction, and subsequent analysis of the odour-active volatiles. Observed changes in their odour intensity, including the formation of some off-flavours such as methional, furaneol, 4-vinylguaiacol and guaiacol, were not significant to that extent to lead to an evident worsening in the overall flavour of juices. Thus, the use of nitrogen atmosphere proved its ability to protect the organoleptic quality of juice from undesirable changes caused by oxidative load or acid-catalysed reactions. Aroma profiles of fresh juices were considerably influenced by diverse climatic conditions, and different seasons of orange harvest in monitored years. Pasteurization and storage had lesser impact on the volatiles in 2018 as in 2019, probably due to the inter-annual variability in such parameters of juices as content of pulp and pH.

Keywords Orange juice · Storage · Modified pasteurization · Volatiles · Solid-phase microextraction · Gas chromatography–olfactometry

Introduction

As the main product of orange processing, orange juice is appreciated by consumers all over the world. Its high market share can be attributed to its unique, a widely favoured aroma, colour and health benefits. However, these qualities of orange juice, besides the influence of climatic conditions, can vary because of differences in applied processing technologies as well as diverse storage conditions. In recent years increases demand of consumers for juice products with properties as close as possible to those of freshly pressed unpasteurized juices (Bi et al. 2020). Thus, main challenges for the producers of orange beverages are a gentle juice processing with minimal impacts on its nutritional and sensory properties, the guarantee of this quality as well as microbial safety during shelf life, that can be up to several months (Mastello et al. 2015).

One of the most commonly used processing techniques in food industry, which were developed to achieve shelf-stable products, is pasteurization by heat. It is still the most cost-effective method, discovered to date for reducing the microbial contamination and enzymatic activities in juice matrix. However, this process may change the quality of orange juice in terms not only of nutritional ingredients, but also can cause the loss of desirable aroma compounds, and/or to initiate the formation of undesirable off-flavours (Bi et al. 2020; Kim et al. 2018; Mastello et al. 2015).

Farnworth et al. (2001) compared the content of volatiles of pasteurized and frozen unpasteurized orange juice after storage. Concerning the unpasteurized juice, they found higher concentrations of acetaldehyde, ethyl acetate,

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α-terpineol, 1-hexanol, 3-hexen-1-ol, α-pinene, sabinene, β-myrcene and limonene but, on the contrary, lower concentration of valencene. The authors concluded that unpasteurized juice, which is quickly frozen and kept frozen until use, may be more acceptable by the consumer, who is looking for a fresh squeezed juice rather than for pasteurized one. In study of Sádecká et al. (2014), a decrease mainly in the contents of some terpene alcohols, esters, aldehydes, ketones and sesquiterpenes, together with an increase in the content of some volatile monoterpenes were observed after pasteurization of orange juice. On the contrary, according to Jordán et al. (2003), the biggest changes in the content of volatile components occurred during deaeration, the pasteurization process did not modify the composition of deaerated orange juice in a significant way.

Therefore, alternatives to a traditional thermal processing which do not involve direct heating were investigated in order to obtain safe products but with fresh-like quality attributes. Among these, high pressure (HP) and pulsed electric field (PEF) processing received attention. Vervoort et al. (2012) compared these methods with a mild thermal treatment of orange juice. Their results demonstrated that when processing conditions are selected based on equivalent microbial safety, the impact of heat, HP and PEF pasteurization on the volatile profile of orange juice can be considered comparable. On the contrary, in later study of Bi et al. (2020), the comparison of pasteurization and HP treatment revealed compounds that could serve as discriminant indicators of these methods, such as heptanal, (*E*)-2-heptenal, (*E*)-2-nonenal and *d*-carvone for HP treatment, and β-terpineol, *p*-mentha-1,5-dien-8-ol, carveol and β-copaene for pasteurization. Moreover, *d*-carvone and β-terpineol could be used as discriminant indicators throughout the storage period. Mastello et al. (2018) evaluated volatiles and sensory acceptance of non-processed orange juice compared with HP processed and pasteurized juices. Statistical analysis proved differences in composition of processed and non-processed juices, as well as HP treated and pasteurized juices. However, sensory acceptance was similar for HP and pasteurized orange juice.

Additional changes in juice composition can occur during its storage in a retail chain. The number and type of physico-chemical reactions and their interactions make the quantitative study of quality degradation of orange juice during shelf life highly complex. Moreover, storage conditions such as temperature, time, oxygen content, light exposure and packaging material can significantly affect the extent of these reactions (Wibowo et al. 2015).

Regarding the temperature, Petersen et al. (1998) proved that storage of orange juice at 5 °C, as compared with higher temperatures, prevents both sensory and concentration changes of aroma compounds. Storage at ambient temperature led to an increase in concentrations of α- and β-terpineol, which contribute to the oxidized aroma and bitterness of juice. Similarly, Moshonas and Shaw (1989) observed a decrease in concentrations of 1-penten-3-one, hexanal, ethyl butanoate, octanal, neral and geranial, and an increase in concentrations of some undesirable components such as furfural and α-terpineol, during storage of aseptically packed orange juice. The increase in contents of other well-known off-flavour compounds, in particular dimethyl sulphide, 4-vinylguaiacol, α-terpineol and furaneol, was revealed by Averbeck and Schieberle (2011) during forced and normal storage of reconstituted orange juice. Wibowo et al. (2015) studied chemical reactions taking place in pasteurized orange juice during storage at room and elevated temperatures. The most significant changes were observed in group of terpenes and in oxides and sulphur compounds. In addition, decrease in content of aldehydes and esters was observed. At the same time, the authors proposed the compounds such as α-pinene, α-terpineol, linalool and octanal as potential markers of pasteurized and stored orange juice.

In majority of reactions ongoing in food products, oxygen can play an important role and thus barrier properties of the packaging material against oxygen diffusion from the surrounding environment is the next often studied parameter, which is supposed to affect the stability of juice during storage. However, in the study of Berlinet et al. (2005), the same evolution in the content of volatiles was observed during the storage of orange juice, regardless of the type of used packaging material—glass, and three kinds of polyethylene terephthalate (PET). Used PET packaging materials and their related values of oxygen permeability showed no correlation with the loss of aroma compounds. These findings were confirmed by Bacigalupi et al. (2013), who investigated sensitivity of orange juice to oxidation, when the standard PET or active PET bottles with oxygen scavengers were used. The aroma changes were not relevant markers of oxygen ingress and permeability properties of the packaging materials, suggesting that the modification of the aroma profile during storage was caused mainly by acid-catalysed reactions, and only to a lesser extent by oxidation. The role of acid-catalysed reactions in the degradation of juice was supported also by another study (Berlinet et al. 2006) in which the rise in pH from 3.2 up to 4.0 significantly reduced the concentrations of off-flavours furfural and α-terpineol during storage. Despite of above mentioned primary role of acid-catalysed reactions in the degradation of orange juice, our previous study (Kopuncová et al. 2018) dealing with impact of inert gas application on the profile of orange juice volatiles during 4-month storage of juices revealed followed findings: the changes taking place in juice samples which were processed in nitrogen (N_2) as well as carbon dioxide (CO_2) atmosphere at only certain technological stages of their production, were not significant sensorially, and they

did not lead to noticeable deterioration of organoleptic properties of juices during the storage. On the contrary, negative sensory changes in flavour of stored juices were observed for juices processed in conventional "air" atmosphere. They could be caused by the increase in content of some aldehydes during the storage, as a consequence of oxidative changes which are going on in juice matrix. Mentioned worsening of organoleptic properties manifested itself mainly in increased bitter and astringent taste of juice, a certain loss of freshness and fruity sweetness, and undesirable colour changes.

To the best of our knowledge, the studies aimed at comprehensive evaluation of continuous inert gas application (used in all technological stages of juice processing) on the aroma stability, and sensory qualitative attributes of industrially produced fruit juices are still missing. Therefore, based on the results of our previous work (Kopuncová et al. 2018), we focused this study on the monitoring of effects of modified gentle pasteurization, and exactly defined 4-month storage on the odour stability of principal aroma-active compounds revealed by the method of gas chromatography–olfactometry (GC-FID/O). The use of this specific combined sensory and instrumental technique allowed us to provide complex evaluation of this type of juice, completely processed under N_2 atmosphere, not only from analytical but mainly from organoleptic point of view. Moreover, two series of storage experiments were carried out throughout two following years with the aim to compare the effect of inter-annual differences on the quality of juice.

Materials and methods

Samples and storage conditions

Samples of orange juice enriched with pulp were obtained from McCarter, Bratislava, Slovakia (production premises Dunajská Streda, Slovakia). This company imports raw unconcentrated juice in frozen state from several countries of origin, in this case from Costa Rica. After unfreezing, juice was enriched with pulp, mixed, gently pasteurized at 95 °C during 20 s and filled aseptically into 200 mL PET bottles with oxygen scavengers. Juice was fully processed and packed under inert N_2 atmosphere. Bottled samples were stored in the refrigerator at 7 ± 1 °C in darkness, within 4-month shelf life period. Analyses of raw unpasteurized and fresh pasteurized juice were performed within 24 h after delivery of samples to the laboratory. Other samples of pasteurized juice were subsequently analysed on a monthly basis. The effects of pasteurization and storage on the volatiles of orange juice were investigated over two following production years (2018, 2019).

Chemicals

All chemicals used as reference standards for identification purposes of volatiles (listed in Tables 1 and 2) were gifts donated from Bedoukian Research (Danbury, Connecticut, USA), Graz University of Technology (Graz, Austria) and French National Institute for Agricultural Research (INRA) laboratories (Dijon, France).

Methods

Headspace solid-phase microextraction

The method was selected for the purpose of extraction of orange juice overall volatile fractions containing aroma-forming compounds. Each sample of juice (5.0 mL) was incubated statically in a 40 mL glass vial in a metallic block thermostat (Liebisch, Bielefeld, Germany) at 35 °C for 30 min. Isolation of volatiles was carried out by a solid-phase microextraction (SPME) fibre placed in the headspace of the sample during the entire incubation period. The SPME fibre with divinylbenzene/Carboxen/polydimethylsiloxane (DVB/Carboxen/PDMS) film (2 cm), film thickness 50/30 µm, "For odours" (Supelco, Bellefonte, Pennsylvania, USA) was used. The fibre was initially conditioned by heating in the injector block of gas chromatograph at 270 °C for 1 h. Headspace solid-phase microextraction (HS-SPME) samples were desorbed at 250 °C in the injector block of the gas chromatograph during the entire GC analysis.

Gas chromatography–olfactometry

Complex mixtures of volatile compounds extracted by HS-SPME were separated and analysed by gas chromatography coupled to flame ionization detection and olfactometry (GC-FID/O) using the detection frequency concept of posterior evaluation of odour quality and odour intensity of individual odorants, according to the modified procedure of Janáčová et al. (2008). A sniffing procedure panel was formed from 3 judges (2 women, 1 man, aged 29–57 years), who were chosen from 5 assessors trained in sensory evaluation. Results of GC-FID/O analyses were expressed as average values of estimated odour intensities in a scale from 0 to 3 with increments of 0.5, obtained from 6 independent measurements for each sample, complying with the requirement of at least 5 citations within each olfactory percept. The value ± 0.5 was considered as a standard error of estimation of odour intensities for applied intensity scale and engaged well trained sensory panel. For the performance of these analyses, the gas chromatograph Agilent 7890A (Agilent Technologies, Palo Alto, California, USA) was coupled to flame ionization detector (FID)

Table 1 Odour-active compounds of raw, pasteurized and stored orange juice processed in 2018 revealed by HS-SPME coupled to GC-FID/O and their related odour intensities

No.	LRI[DBW]	Odour intensity						Compound	Odour description	References
		Raw juice	Past. juice	1 month	2 months	3 months	4 months			
1	893.1	–	–	–	–	1	1	Ethylacetate	Ethereal, sweetish, fruity	LRI, MS, OD, LIT
2	–	–	0.5	0.5	0.5	0.5	–	Unknown	Fruity, pleasant	–
3	910.9	–	–	0.5	0.5	0.5	0.5	Methyl propanoate	Pleasant, fruity	LRI, MS, OD, LIT
4	–	1	1	1	0.5–1	0.5	0.5	Ethanol	Alcoholic, ethereal	MS, OD, LIT
5	979.7	0.5	0.5	0.5	0.5	0.5	–	2-Pentanone	Weak fruity	LRI, OD, LIT
6	1020.9	0.5–1	0.5–1	1	1	0.5	0.5	α-Pinene	Terpeny, sharp, fresh, pine	LRI, MS, OD, LIT
7	1040.4	1	1.5	2	2	1.5	1.5	Ethyl butanoate	Fruity, fragrant, apple-like, sweet	LRI, MS, OD, LIT
8	1055.2	1	1	1	1	2	2	Ethyl 2-methylbutanoate	Sweet, ripened fruit-like, pungent	LRI, MS, OD, LIT
9	–	–	–	0.5	1	1.5	2	Isoterpinolene	Fresh, green, plant	MS, OD, LIT
10	–	0.5	0.5	0.5	0.5	1		Unknown	Fresh, plant-like	–
11	1085.2	1	1	1	0.5–1	0.5	0.5	Hexanal	Green, plant-like, fresh, slight fruity	LRI, MS, OD, LIT
12	1147.8	1	1	1.5	2	0.5–1	0.5–1	δ-3-Carene	Fresh, terpenic, fruity, sweetish citrus, coniferous	LRI, MS, OD, LIT
13	1167.3 + 1179.4	1	1.5	1.5	2	0.5–1	0.5–1	Myrcene + α-terpinene	Fresh green plant-like, herbaceous, terpenic, balsamic	LRI, MS, OD, LIT
14	1200.0 + –	2.5	2.5	2.5	2.5	3	3	Limonene + β-phellandrene	Fresh, orange-like, citrus peel-like, bitterish + terpenic, minty	LRI, MS, OD, LIT
15	1241.2	1.5	1.5	1.5	2.5	2		Ethyl hexanoate	Sweet, fruity, brandy-like	LRI, MS, OD, LIT
16	1247.1	1	1	1		1.5	2	γ-Terpinene	Citrus-like, herbaceous	LRI, MS, OD, LIT
17	1256.0	0.5	0.5	1		1.5	2	(*E*)-β-Ocimene	Citrus-like	LRI, MS, OD, LIT
18	1272.4	1	0.5–1	1	1	0.5–1	0.5–1	*p*-Cymene	Pleasant, fresh, citrus, lemon	LRI, MS, OD, LIT
19	1284.6	1	1	1	1	0.5–1	0.5–1	α-Terpinolene	Sharp terpenic, fuel-like, plastic, petroleum	LRI, MS, OD, LIT
20	1293.0	1.5	1.5	2	1	0.5	0.5	Octanal	Citrus peel-like, fatty, waxy, slight bitterish	LRI, MS, OD, LIT
21	–	–	0.5	–	1	–	–	(*E*)-4,8-Dimethyl-1,3,7-nonatriene	Fine flowery scent	MS, OD, LIT
22	1334.2	–	–	1	1	0.5	0.5	3-Methyl-2-butenol	Fresh, fruity, green note, cowberry-like	LRI, MS, OD, LIT
23	1381.1	0.5	0.5	0.5	0.5–1	–	–	Heptyl acetate	Fragrant, citrus, fruity	LRI, MS, OD, LIT
24	1397.7	1.5	1	1	1	0.5	0.5	Nonanal	Waxy, fresh fruity, sharp, aldehydic irritant odour	LRI, MS, OD, LIT
25	1442.3	1.5	1.5	1.5	2	2	2.5	Ethyl octanoate	Strong fruity, sweet, orange-like, floral, balsamic	LRI, MS, OD, LIT
26	1456.0	–	–	0.5–1	0.5–1	–	–	Methional	Cooked potato-like	LRI, OD, LIT
27	–	–	0.5	–	0.5–1	–	–	*p*-Propenyl toluene	Spicy, guaiacol-like, slight orange peel-like	MS, OD, LIT
28	1483.1	1	1	1.5	1.5	1	1	Octyl acetate	riPened citrus fruit, sweet orange juice-like	LRI, MS, OD, LIT
29	1489.9	1	1	1	1	0.5	0.5	α-Copaene	Woody, spicy, slight honey	LRI, MS, OD, LIT
30	1502.9	1	1	1	1	0.5	0.5	Decanal	Sweetish, citrus, fresh with fatty and astringent undertones	LRI, MS, OD, LIT

Table 1 (continued)

No.	LRI^{DBW}	Odour intensity						Compound	Odour description	References
		Raw juice	Past. juice	1 month	2 months	3 months	4 months			
31	1520.9	0.5	0.5	0.5	0.5–1	1	0.5–1	Benzaldehyde	Fresh, bitterish, cherry-like, almond-like	LRI, MS, OD, LIT
32	1559.9	2.5	2.5	2	2.5	2	2	Linalool	Pleasant, gentle fresh, citrus-like, floral odour	LRI, MS, OD, LIT
33	1571.7 + –			1	1	1		Octanol + limona ketone[t]	Waxy, citrus-like, herbal undertone	LRI, MS, OD, LIT
34	–	0.5	0.5	0.5	0.5	0.5	–	β-Copaene	Pleasant, gentle odour, slight sweetish, fruity	MS, OD, LIT
35	1592.9 + 1609.5	0.5	0.5	0.5	0.5	1	1.5	β-Caryophyllene + terpinen-4-ol	Terpene odour, spicy, nutmeg-like, woody	LRI, MS, OD, LIT
36	1641.2	0.5	0.5	0.5	0.5			β-Terpineol	Burnt, earthy, exhaust fumes-like	LRI, MS, OD, LIT
37	1674.3	1	0.5	0.5	0.5–1	1	1–1.5	Nonanol	Sweetish, slight tobacco-like, weak floral, citrus freshness	LRI, MS, OD, LIT
38	–	–	1	1	1	1	1	Unknown	B-vitamins complex, meat consommé-like, sweetish	–
39	1705.6	0.5–1	1	1	1.5	1.5	1.5	α-Terpineol	Heavier sweetish floral odour, wild rose-like	LRI, MS, OD, LIT
40	1717.1	0.5–1	1	1	1	1.5	1.5	Valencene	Fresh-sweet juicy orange, orange peel-like, slight waxy	LRI, MS, OD, LIT
41	–	0.5	1	1–1.5	1–1.5		1	α-Selinene	Fresh, citrus-like, floral, spice essential oil-like	MS, OD, LIT
42	1727.3	0.5	0.5	0.5	0.5	0.5	0.5	Methionol	Cooked potato-like, slight meaty bouillon-like	LRI, OD, LIT
43	1734.0	–	0.5	–	0.5	0.5	–	Neryl acetate	Fruity, orange-like	LRI, MS, OD, LIT
44	1774.2 + –	0.5	0.5	0.5	1	0.5	–	Methyl salicylate + perillaldehyde	Herbal, fresh, minty, citrus-like	LRI, MS, OD, LIT
45	1812.6	0.5–1	0.5–1	1.5	1	1	1	Nerol	Sweet rose, citrus undertone	LRI, OD, LIT
46	1859.0	0.5–1	0.5–1	1.5	1.5	1.5	0.5–1	Guaiacol	Smoked odour	LRI, OD, LIT
47	1860.5	1	1				2	Geraniol	Intensive balsamy odour, heavier sweetish rose odour	LRI, OD, LIT
48	1914.7	1	1	1	1	1.5	1.5	γ-Octalactone	Fragrant, coconut-like, creamy, sweet, fatty	LRI, OD, LIT
49	1917.9	0.5	1	1	1	1	1	2-Phenyl ethanol	Rose-like, heavier sweetish odour	LRI, OD, LIT
50	2043.3	–	1	1	1	2	3	Furaneol	Caramel-like	LRI, OD, LIT
51	–	–	0.5	0.5	0.5	1		Octanoic acid	Weak warm oily-like odour, slight musty heavier odour	MS, OD, LIT
52	2192.0	–	0.5	1	1	1	2.5	4-Vinylguaiacol	Maggi, spice-like	LRI, MS, OD, LIT
53	2194.6	1	1.5	1.5	1.5	2	2	δ-Decalactone	Creamy, coconut, sweetish	LRI, MS, OD, LIT
54	–	0.5	0.5	0.5	0.5	0.5	–	Decanoic acid	Fatty, citrus-like undertone	MS, OD, LIT
55	–	0.5	1	1	1	1	1	Unknown	Pleasant, fresh, fragrant herb-like	–
56	–	–	–	0.5–1	0.5–1	1	1	Unknown	Weak pleasant odour, gentle herbal	–

Compounds were identified on the basis of following criteria: LRI^{DBW} linear retention index measured on GC column DB-WAX; *MS* mass spectrum; *OD* odour quality; *LIT* literature reference. *t* tentative identification (only on the basis of mass spectra)

Table 2 Odour-active compounds of raw, pasteurized and stored orange juice processed in 2019 revealed by HS-SPME coupled to GC-FID/O and their related odour intensities

No.	LRI[DBW]	Odour intensity						Compound	Odour description	References
		Raw juice	Past. juice	1 month	2 months	3 months	4 months			
1	893.1	1	1	1	1	1	1	Ethylacetate	Ethereal, sweetish	LRI, MS, OD, LIT
2	904.8	0.5	0.5	0.5	0.5	0.5	0.5	Isopropyl acetate	Ethereal, fruity, sweet, pleasant	LRI, MS, OD, LIT
3	–	0.5	0.5	0.5	1	1	1	Ethanol	Alcoholic, ethereal	MS, OD, LIT
4	979.7	1	1	1	1	0.5	0.5	2-Pentanone	Weak fruity, sweetish	LRI, OD, LIT
5	–	0.5	0.5	0.5–1	1	1	1	Unknown	Fruity, sweet	–
6	1020.9	1	1	1	1	1	1	α-Pinene	Terpeny, sharp, fresh, pine	LRI, MS, OD, LIT
7	1040.4	2	2	2	2	2	2	Ethyl butanoate	Fruity, fragrant, apple-like, sweet, orange-like	LRI, MS, OD, LIT
8	1055.2	–	1.5	–	–	0.5	1	Ethyl 2-methylbutanoate	Sweet, ripened fruit-like, pungent	LRI, MS, OD, LIT
9	–	0.5	0.5	–	–	0.5	0.5	Unknown	Sweetish, fruit	–
10	–	1.5	1.5	1.5	1.5	1.5	1.5	Isoterpinolene	Fresh, green, plant	MS, OD, LIT
11	1062.8	–	0.5	0.5	0.5	0.5	1	Camphene	Camphorous, cooling, fresh, citrus, pine, green	LRI, MS, OD, LIT
12	1085.2	0.5	0.5	0.5	–	–	0.5	Hexanal	Green, plant-like, fresh, slight fruity	LRI, MS, OD, LIT
13	1147.8	0.5	0.5	0.5	–	–	–	δ-3-Carene	Fresh, terpenic, fruity, sweetish citrus, coniferous	LRI, MS, OD, LIT
14	1167.3 + 1179.4	1.5	1.5	1.5	1.5	1.5	1.5	Myrcene + α-terpinene	Fresh green plant-like, herbaceous, terpenic, balsamic	LRI, MS, OD, LIT
15	–	0.5	0.5	0.5	–	–	–	4-carene	Weak warm, dry	MS, OD, LIT
16	1200.0 + –	2.5	2.5	2.5	2.5	2.5	2	Limonene + β-phellandrene	Fresh, orange-like, citrus peel-like, bitterish + terpenic, minty	LRI, MS, OD, LIT
17	1241.2	2	2	2	2	2		Ethyl hexanoate	Sweet, fruity, brandy-like	LRI, MS, OD, LIT
18	1247.1	1.5					2	γ-Terpinene	Citrus-like, herbaceous	LRI, MS, OD, LIT
19	1256.0	1.5						(*E*)-β-Ocimene	Sweet, fruity, pleasant	LRI, MS, OD, LIT
20	1272.4	–	–	1				*p*-Cymene	Pleasant, fresh, citrus, lemon	LRI, MS, OD, LIT
21	1284.6	0.5	1	1	1.5	1.5	1	α-Terpinolene	Sharp terpenic, fuel-like, plastic, petroleum	LRI, MS, OD, LIT
22	1293.0	2	2	2	2	2	2	Octanal	Citrus peel-like, fatty, waxy, slight bitterish	LRI, MS, OD, LIT
23	1306.5	–	–	1	1	1	1	1-Octen-3-one	Mushroom-like, earthy	LRI, OD, LIT
24	–	1	1	–	1	1	–	(*E*)-4,8-Dimethyl-1,3,7-nonatriene	Sweetish, fine flowery	MS, OD, LIT
25	1334.2	–	–	1	1	0.5–1	0.5	3-Methyl-2-buten-1-ol	Fresh, fruity, green note, cowberry-like	LRI, MS, OD, LIT
26	1381.1	0.5	0.5	0.5	0.5	–	–	Heptyl acetate	Fragrant, citrus, fruity	LRI, MS, OD, LIT
27	1397.7	1.5	1.5	1	1	1	0.5	Nonanal	Waxy, fresh, citrus peel-like, sharp, aldehydic irritant odour	LRI, MS, OD, LIT
28	1442.3	1.5	1.5	2	2	2	2	Ethyl octanoate	Strong fruity, sweet, orange-like, floral, balsamic	LRI, MS, OD, LIT
29	1456.0	–	–	–	0.5	0.5	–	Methional	Earthy, cooked potato-like	LRI, OD, LIT

Table 2 (continued)

No.	LRI[DBW]	Odour intensity						Compound	Odour description	References
		Raw juice	Past. juice	1 month	2 months	3 months	4 months			
30	–	0.5	0.5	0.5	0.5	0.5	1	*p*-Cymenene	Spicy, guaiacol-like, slight orange peel-like	MS, OD, LIT
31	1483.1	1.5	1.5	1.5	1.5	1.5	1.5	Octyl acetate	Ripened orange fruit, sweet orange juice-like	LRI, MS, OD, LIT
32	1489.9	1	1	1				α-Copaene	Woody, spicy, slight honey	MS, OD, LIT
33	1502.9	1	1	1	1		0.5	Decanal	Sweetish, citrus, fresh with fatty and astringent undertones	LRI, MS, OD, LIT
34	1520.9	0.5	–	–	0.5	–	0.5	Benzaldehyde	Fresh, bitterish, cherry-like, almond-like	LRI, MS, OD, LIT
35	1559.9	2.5	2	2	2	2	2	Linalool	Pleasant, gentle fresh, citrus-like, floral odour	LRI, MS, OD, LIT
36	1571.7 + –	1.5	2	2	2.5	2	2	Octanol + limona ketone[t]	Waxy, citrus-like, herbal-spicy undertone	LRI, MS, OD, LIT
37	–	0.5	0.5	–	0.5	0.5	–	β-Copaene	Pleasant, slight sweetish, fruity	MS, OD, LIT
38	1609.5	1	1	1.5	1.5	1.5	1.5	Terpinen-4-ol	Pleasant, spicy, nutmeg-like, woody	LRI, MS, OD, LIT
39	1641.2	1	1	1				β-Terpineol	Earthy, exhaust fumes-like	LRI, MS, OD, LIT
40	1674.3	1	1	1	1	1	1	Nonanol	Sweetish, slight tobacco-like, weak floral, citrus freshness	LRI, MS, OD, LIT
41	–	1	1	–	–	–	0.5	Unknown	Sweetish, B-vitamins complex-like, meat consommé-like	–
42	–	0.5	0.5	0.5	0.5	0.5	0.5	γ-Muurolene	Woody, herbal, spicy	MS, OD, LIT
43	1705.6	0.5	1	1	1	1	1.5	α-Terpineol	Heavier sweetish floral odour, lilac-like	LRI, MS, OD, LIT
44	1717.1	0.5–1	0.5–1	1			1	Valencene	Fresh-sweet juicy orange, orange peel-like, slight waxy	LRI, MS, OD, LIT
45	–	0.5–1	0.5–1	1	1	1	1	α-Selinene	Fresh, citrus-like, floral, spice essential oil-like	MS, OD, LIT
46	1734.0	–	1	1	1	1	1	Carvone	Fresh, minty, herbal	LRI, MS, OD, LIT
47	1776.4	1	1	1	1	1	1	Decanol	Fruity, plum compote-like	LRI, MS, OD, LIT
48		1	1	1	1	1	1	perillaldehyde	Fresh, minty, citrus, fragrant, herbal odour	MS, OD, LIT
49	1859.0	1.5	1.5	1.5	1.5	1.5	1.5	Guaiacol	Smoked odour	LRI, OD, LIT
50	1860.5	1						Geraniol	Intensive balsamy odour, heavier sweetish rose odour	LRI, MS, OD, LIT
51	1914.7	1	1	1	1	1	1	γ-Octalactone	Fragrant, coconut-like, creamy, sweet, fatty	LRI, OD, LIT
52	1917.9	0.5	0.5	0.5	0.5	0.5	–	2-Phenyl ethanol	Rose-like, heavier sweetish odour	LRI, OD, LIT
53	2043.3	–	1	1	1.5	1.5	2	FURANEOL	Caramel-like	LRI, OD, LIT
54	–	0.5	0.5	0.5	1	1	1	Octanoic acid	Warm oily-like odour, fatty, slight musty heavier odour	MS, OD, LIT
55	2192.0	–	1	1	1	1	1	4-Vinylguaiacol	Maggi, curry spice-like	LRI, MS, OD, LIT
56	2194.6	1	1	1	1	1	1	δ-Decalactone	Creamy, coconut, sweetish	LRI, MS, OD, LIT
57	–	0.5	0.5	1	1	1	1	Decanoic acid	Soap-like, fatty, waxy, citrus undertone	MS, OD, LIT

Table 2 (continued)

No.	LRI^{DBW}	Odour intensity						Compound	Odour description	References
		Raw juice	Past. juice	1 month	2 months	3 months	4 months			
58	–	0.5–1	0.5–1	0.5–1	1	1	1	Unknown	Pleasant, fresh, fragrant herb-like	–
59	–	0.5–1	1	1	1	1	1	Unknown	Weak pleasant odour, gentle herbal	–

Compounds were identified on the basis of following criteria: LRI^{DBW} linear retention index measured on GC column DB-WAX; *MS* mass spectrum; *OD* odour quality; *LIT* literature reference. *t* tentative identification (only on the basis of mass spectra)

and an olfactory detection port (ODP), ODP3 (Gerstel, Mülheim an der Ruhr, Germany). The capillary GC column DB-WAX (30 m × 0.32 mm × 0.25 µm; Agilent Technologies) operated with a temperature programme 35 °C (1 min), 5 °C/min, 200 °C (1 min). Hydrogen was used as a carrier gas at the linear velocity of 44.6 cm/s (measured at column temperature 143 °C). Pulsed splitless injection mode was used at injector temperature of 250 °C. For GC-FID/O experiments, the effluent of the column was splitted with a split ratio of 1:1 to FID and the olfactory (sniffing) port ODP with addition of humidified air to protect the nose epithelium from dehydration. FID temperature was set to 250 °C. ODP operated at a temperature of 180 °C, an interface temperature was 230 °C, and the flow of added nitrogen in ODP humidifier was 12 mL/min. The sniffing time of each judge did not exceed 30 min.

Gas chromatography-mass spectrometry

In parallel with GC-FID/O, HS-SPME extracts of volatiles were analysed by gas chromatography-mass spectrometry (GC–MS) using the gas chromatograph Agilent 6890N (Agilent Technologies, Palo Alto, California, USA) coupled to the mass spectrometric detector 5973 inert (Agilent Technologies) equipped with capillary column DB-WAXetr (30 m × 0.25 mm × 0.50 µm; Agilent Technologies) operating with a temperature programme 35 °C (1 min), 5 °C/min, 220 °C (1 min). The linear velocity of carrier gas helium was 45 cm/s (measured at 143 °C). Pulsed splitless injection mode was used at an injector temperature of 250 °C. Ion source operated at a temperature of 230 °C and quadrupole at a temperature of 150 °C. Ionization voltage (EI) was set to 70 eV.

Identification of volatile compounds

The volatiles were identified on the basis of comparison of their linear retention indices, mass spectra, GC analysis of standards, and by the comparison of data on occurrence and odour description with literature. The linear retention indices (LRI) were calculated using the equation of Van den Dool and Kratz (1963) and standard mixture of *n*-alkanes C_8–C_{22} was used as reference. LRI data were compared and confirmed with LRI data obtained by measurement of standard volatile compounds. For this purpose, our in-house database of LRI data was used. Identification of compounds was performed additionally by comparison of measured mass spectra with available mass spectral libraries Wiley and NIST MS (National Institute of Standards and Technology, Gaithersburg, Maryland, USA).

Statistical analysis

Statistical calculations were performed by means of Unistat v. 6.0 (Unistat, London, United Kingdom) statistical package. Principal component analysis (PCA) was used in order to define, interpret and visualize the differences between the compared orange juice samples produced in 2 years as well as to assess the effects of pasteurization and storage.

Results and discussion

GC-FID/O study of orange juices produced in 2018 and 2019

In general, 61 key odour-active compounds were detected in the orange juice produced in 2018 (Table 1). However, due to the overlaps between odours of individual volatiles in four cases (β-myrcene + α-terpinene, limonene + β-phellandrene, octanol + limona ketone[t], β-caryophyllene + terpinen-4-ol), only 56 olfactory responses were recorded. Fifty-five compounds were identified by a combination of independent methods as indicated in "Materials and methods" section. In case of limona ketone only partial information was available and thus, only tentative identification was possible. Five compounds remained unidentified at this stage because they were detected only by GC–olfactometry. In juice produced in 2019 (Table 2) the comparable number of key odorants (62), as well as olfactory responses (59, because of overlaps of odours of β-myrcene + α-terpinene, limonene + β-phellandrene, octanol + limona ketone[t]) were recorded. Fifty-six compounds were completely identified, in case of limona ketone only tentative identification was possible again, and five compounds remained unidentified.

The overall aromas of analysed juices were found to be formed by the components that belong to different chemical groups. There were identified seventeen terpenes, six terpene alcohols, six aldehydes, six alcohols, ten esters, one ketone in juice samples from 2018, and twenty-one terpenes and five terpene alcohols, seven aldehydes, six alcohols, eight esters, three ketones in juice samples from 2019. Two lactones, two carboxylic acids as well as one derivative of furan and two phenol derivatives were identified in juice samples from both years. In comparison with results of our previous study (Kopuncová et al. 2018), the aroma profile of samples investigated in current study was composed from much greater number of odour-active compounds. This was caused by using GC column with polar stationary phase, while in our previous work GC column with nonpolar phase was used.

With respect to the measured odour intensities, the principal odour-active compounds of volatile fractions of juices produced in both years were limonene + β-phellandrene, linalool, ethyl butanoate, ethyl hexanoate, ethyl octanoate, octanal and octanol + limona ketone[t]. In 2019, all these compounds showed high odour intensities from 2 to 2.5. In 2018, they had lower intensity 1.5, except for limonene + β-phellandrene and linalool. Other aroma-impacting compounds (odour intensity 1 in 2018 and 1.5 in 2019) were isoterpinolene, β-myrcene + α-terpinene, γ-terpinene, (*E*)-β-ocimene, nonanal and octyl acetate. Several studies were focused on the GC-O characterization of volatile fraction of orange juice, in which different compounds were identified as most sensorially important. Tønder et al. (1998) identified ethyl butanoate, β-pinene, limonene, octanal and linalool to be the most important odorants in fresh and stored orange juice by the calculation of aroma values, as well as GC-sniffing technique called GC Odour Profiling. Averbeck and Schieberle (2009) revealed linalool, limonene, ethyl 2-methylbutanoate, octanal, α-pinene, β-myrcene, acetaldehyde, decanal and β-damascenone as volatiles with the highest odour activity values in the aroma of freshly reconstituted orange juice from concentrate. In a comparative study of different orange varieties, Arena et al. (2006) observed the highest frequency of odour detection for ethyl butanoate, α-pinene and β-myrcene in juices from blond orange varieties. Comparison of our above mentioned findings and results presented in these studies showed some differences. However, these could be explained by different ways of orange juice production, fruit varieties, and techniques used to collect and process GC-O data.

As regards the pasteurization, GC-O study did not show significant changes in odour intensities of individual odorants. Slight increase was observed for some terpenes and alcohols such as β-myrcene + α-terpinene (from 1 to 1.5), α-terpineol, valencene and α-selinene (from 0.5 to 1 for all three compounds) in 2018, and for γ-terpinene and (*E*)-β-ocimene (from 1.5 to 2 for both compounds), α-terpinolene and α-terpineol (0.5 to 1 for both compounds) as well as for octanol + limona ketone[t] (from 1.5 to 2) in 2019. On the contrary, slight decrease of odour intensity of linalool (from 2.5 to 2) was observed during thermal treatment of juice in 2019. In 2018, decrease of linalool was not observed but there was noticed slight decrease of nonanal (from 1.5 to 1) and nonanol (from 1 to 0.5). The increase in odour intensities of terpene alcohols after heat treatment, such as α-terpineol in our study, could mainly originated from the series of oxidative hydration-dehydration reactions of hydrocarbon terpenes and other precursors (Perez-Cacho and Rouseff 2008). Compounds α-terpineol, as well as terpinen-4-ol were previously reported (Bazemore et al. 1999) in excessively heated orange juice, and they are commonly considered to be thermally generated off-flavours, while they impart stale and musty notes to orange juice. Bi et al. (2020) observed the increase in the content of almost all alcohols after pasteurization of orange juice, among which

β-terpineol, *p*-mentha-1,5-dien-8-ol and carveol were not detected in fresh juice, thus they were considered as specific components of thermally processed orange juice. In accordance with our study, Bi et al. (2020) observed also the higher contents of some terpenes such as γ-terpinene, (*E*)-β-ocimene and α-terpinolene in processed juice in comparison with fresh juice. In contrast with our study, some authors observed increase of linalool concentrations after processing of juice (Bazemore et al. 2003; Bi et al. 2020), probably as a results of the release of free linalool from glycosides by heating. In our study, the stable (2018) or only slightly decreasing (2019) odour intensity of linalool after pasteurization of juice could be explained by the fact, that in acidic matrix of orange juice linalool is very easily degraded to other substances, mostly above mentioned terpene alcohols α-terpineol and terpinen-4-ol.

The impact of 4-month storage on the aroma profile of orange juices produced in two following years was not mutually comparable. The differences in the extent of odour intensity changes observed during storage of juices produced in two following years were also confirmed by the statistical analysis of GC-FID/O results that is discussed below in a separate subhead. In samples from 2018, there were observed changes in odour intensities of almost all identified volatiles. There was no compound that would keep stable odour during the entire experiment. The significant increase in odour intensity by 1 to 2 points was noticed for terpene hydrocarbons and alcohols isoterpinolene, γ-terpinene, (*E*)-β-ocimene, β-caryophyllene + terpinen-4-ol, β-terpineol, α-terpineol, valencene, geraniol, some esters as ethyl 2-methylbutanoate, ethyl hexanoate and ethyl octanoate, both lactones γ-octalactone and δ-decalactone, as well as for octanoic acid and nonanol. The decreasing trend showed some terpene hydrocarbons and alcohols such as β-myrcene + α-terpinene, α-terpinolene and linalool, aldehydes hexanal, octanal, nonanal, decanal and ester heptyl acetate. On the contrary, in 2019, the storage of samples did not have such effect on the individual volatiles. There were a lot of compounds which intensity remained stable during the entire experiment, in particular α-pinene, isoterpinolene, β-myrcene + α-terpinene, ethylacetate, ethyl butanoate, octyl acetate, nonanol, decanol, octanal, perillaldehyde, γ-octalactone and δ-decalactone. An increase in odour intensities was observed for terpene hydrocarbons and alcohols camphene, *p*-cymene, α-copaene, α-terpinolene, terpinen-4-ol, α-terpineol, valencene, α-selinene, fatty acids octanoic and decanoic acid, as well as for ethyl octanoate. However, usually it was slight increase by only 0.5 to 1 point of odour intensity. Similarly, slight decreasing trend was observed for limonene + β-phellandrene, heptyl acetate, nonanal, decanal, 2-pentanone and 2-phenyl ethanol. The increase of some terpene alcohols, which were identified also in our study, such as α-terpineol, terpinen-4-ol and β-terpineol, was observed also by other authors (Averbeck and Schieberle 2011; Bacigalupi et al. 2013; Berlinet et al. 2005; Wibowo et al. 2015) as the consequence of acid-catalysed hydration of limonene and linalool, while the rate of their formation is dependent on the pH of juice. Besides these volatiles, increase of other degradative products of linalool such as 1,8-cineole, geraniol and nerol was referred (Perez-Cacho and Rouseff 2008). In our study, moreover, a rise in the formation of geraniol (2018, 2019) and nerol (2018) correlated also with a decrease of linalool (2018) and limonene (2019) during the storage. An increase of some terpene hydrocarbons such as α-terpinolene, α-terpinene and γ-terpinene can be linked to oxidative reaction and acid-catalysed hydration-dehydration reactions of terpenes (Perez-Cacho and Rouseff 2008; Wibowo et al. 2015). Increase of *p*-cymene was described in other studies as a consequence of rearrangement, hydrogenation and dehydrogenation of α-terpinene, γ-terpinene and limonene (Perez-Cacho and Rouseff 2008; Wibowo et al. 2015). Thus, in compliance with appropriate conditions, chemical reactions of particular terpene hydrocarbons can result in the formation and/or degradation of other terpene hydrocarbons. In accordance with our results, the decrease in the content of aldehydes was observed also by Berlinet et al. (2005) and Wibowo et al. (2015), presumably due to conversion into their corresponding acids. The increasing odour intensity of octanoic and decanoic acids in our study confirmed this suggestion. In addition, decrease of saturated aldehydes could be ascribed to their absorption by PET packaging (Wibowo et al. 2015). At the end, it is necessary to mention that there may exist significant differences between the changes in the concentration of volatiles after pasteurization and storage and their real impact on the odour intensity of individual compounds as well as overall aroma of orange juice. Possible discrepancy between the results of GC-O in our study and discussed results of predominately GC–MS analyses from other authors, can be explained by specific features of GC-O technique, e.g. different odour threshold concentrations for olfactory detection of various aroma-active compounds often varying through several orders of magnitude, and/or different psychophysical trend, and/or selectivity of human nose, and/or non-linear response for various kinds of odour-active compounds.

Identification of off-flavours in orange juices produced in 2018 and 2019

Combined technique GC-FID/O can be extremely sensitive for detection of ultra-trace amounts of odour-active compounds, if they have very low odour threshold. This special feature of GC-FID/O allowed us analysis of several volatiles that could not be detected by commonly used instrumental detectors MS and FID, because they were present in samples at very low concentrations but, on the other hand, they were

considerably odour-active. These volatiles represented some well-known off-flavour compounds previously described as processing contaminants of stored orange juice. The first one was methional, which provides cooked potato odour and it is formed by Strecker degradation of L-methionine. Its odour threshold is 0.1–0.2 ng/L in air (Blank et al. 1989). In our study, methional was detected olfactorily for the first time after 1 month of the juice storage in 2018 and after 2 months of storage in 2019. In both years, methional reached only low odour intensity level 0.5–1 in 2018 and 0.5 in 2019. However, these values remained unchanged in both cases only during two storage months, but later, closer to the end of 4-month storage period of juice, they dropped again to zero. Bezman et al. (2001) reported previously increasing concentrations of methional from the fresh to the pasteurized and stored orange juice, and concluded that this compound significantly contributes to the off-flavour of stored juice. Another two identified off-flavours, furaneol and 4-vinylguaiacol, are both products of thermally induced Maillard reactions. Furaneol is formed by reaction of arginine with rhamnose in the presence of ascorbic acid and provides sweet, caramel odour (Haleva-Toledo et al. 1997). Its odour threshold in air is 1 ng/L (Blank and Schieberle 1993). 4-Vinylguaiacol is characterized by the odour of curry pepper and its odour threshold in air is 0.4–0.8 ng/L (Blank et al. 1989). Concerning our study, furaneol really appeared first time in processed juices (2018, 2019) only after their pasteurization at odour intensity level 1 (2018, 2019), and its content and odour intensity continuously increased during the juice storage up to odour value 3 (in 2018) and up to value 2 (in 2019) at the end of the storage. 4-Vinylguajacol appeared first time after pasteurization in both years but, while in 2018 its intensity increased during the storage up to 2.5, in 2019 remained at level 1 during the entire experiment. Similarly, Averbeck and Schieberle (2011) revealed the increase in contents of these compounds during forced as well as normal storage of orange juice reconstituted from concentrate. At the same time, concentration of 4-vinylguajacol clearly exceeded its odour perception threshold, and so confirmed the crucial role of this odorant for the formation of undesirable "stale" off-flavour effect in stored orange juice. On the contrary, concentration of furaneol did not reach its odour perception threshold. The last negative compound identified in our samples was guaiacol, that is a product of metabolism of a common microbial contamination of fruit juices *Alicyclobacillus* spp. and is responsible for medicinal/antiseptic off-flavour of contaminated juices (Gocmen et al. 2005). Its odour threshold in air is 0.1–0.8 1 ng/L in air (Guth and Grosch 1991).

Bianchi et al. (2010) investigated the volatile profile of orange juice contaminated by *Alicyclobacillus* spp. and found that even low levels of contamination can lead to the significant changes in the content of volatiles. Perez-Cacho et al. (2011) established orto- and retro- nasal detection thresholds of guaiacol in orange juice at 0.70 µg/L and 0.53 µg/L, respectively, and its recognition at 2 µg/L. According to our results, in 2018 guaiacol reached only a low odour intensity level 0.5–1. On the contrary, in samples from 2019 was revealed a more significant intensity of guaiacol at level 1.5. However, in both years it was detected also in raw juice, in contrast with other mentioned off-flavour compounds. This indicates, that the contamination of juice did not occur during its processing or storage but already raw material was contaminated during harvesting or squeezing of oranges in the country of origin. This can be also confirmed by unchanging values of guaiacol in all analysed samples of juice in 2018 as well as 2019.

Statistical comparison of GC-FID/O results from 2018 and 2019

The above discussed results of GC-FID/O analyses of both series of orange juice samples were also statistically compared. Variability between the samples from two production years is obvious from results of PCA. Plot of principal components (Fig. 1) clearly indicates the existence of 2 differentiated groups of eigenvectors belonging to the samples from 2018 and 2019. As regards the results of PCA, the first three principal components described more than 70% of the total dataset variability. For description of maximum variability, it would be necessary 10 components with eigenvalue greater than 1. Observed variability could be explained by the diverse climate conditions in individual years, but more significant factor could be different seasons of harvest of oranges because the experiment was carried out from August to December in 2018, and from January to May in 2019.

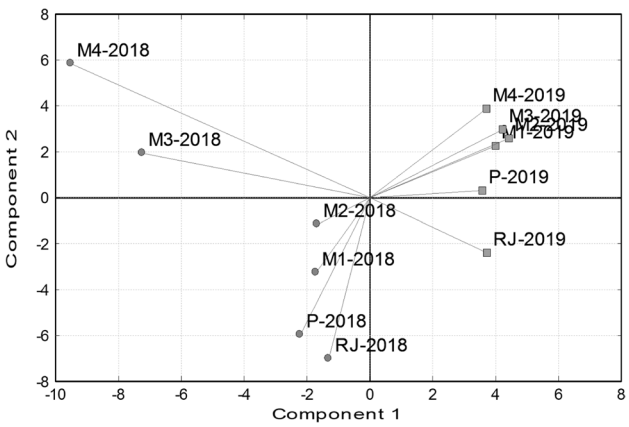

Fig. 1 Plot of principal components demonstrating differentiation of raw, pasteurized and stored orange juices processed in 2018 and 2019 (RJ, raw juice; P, pasteurized juice; M1, M2, M3, M4, juices stored from 1, 2, 3 and 4 months, respectively; plot of principal components was constructed on the basis of odour intensities of individual volatile compounds determined by GC-FID/O

The PCA also demonstrated the significant differentiation of juice samples from individual months of storage in 2018, when the samples stored for three and 4 months were located in different quadrant of the PCA chart than the samples of raw and pasteurized juice, as well as samples stored for 1 and 2 months. On the contrary, in 2019, there was observed similarity between the pasteurized and stored juices because these samples were placed in the same quadrant of the chart in contrast with the sample of raw juice that was placed in the opposite quadrant of the chart. Thus, in 2018, the storage had significantly greater impact on the profile of orange juice odour-active compounds than in the following year with the most significant changes ongoing during third and fourth month of storage. This could be explained by the different pH values of orange juices (3.58 in 2018 and 3.70 in 2019), as well as different contents of fruit pulp (4% in 2018 and 0.12% in 2019) which are parameters that could influence the extent of the acid-catalysed reactions taking place in orange juice samples during the storage.

Conclusions

On the basis of GC-FID/O analyses, it is possible to conclude that some observed changes in the content and odour intensity of some volatiles taking place during pasteurization and storage of the orange juices from both monitored years. These changes relate to mainly the increase of terpene alcohols (α-terpineol, terpinene-4-ol, β-terpineol), loss of some terpene compounds (limonene, linalool) and aldehydes (hexanal, octanal, nonanal, decanal), but they were not sensory significant to that degree, that they would lead to a noticeable deterioration of organoleptic properties of juices. Nevertheless, a certain loss of citrus freshness, as well as typical orange sweetness were noticed in samples at the end of the storage period. As regards the formation of some known off-flavours such as methional, furaneol, 4-vinylguaiacol and guaiacol, that were observed in juices, and they were detected only due to the potential of combined GC-FID/O technique to record ultra-trace amounts of these aroma-active compounds, and of course, as well as due to the sensitivity of the panel of well-trained assessors. However, in the tasting evaluation of stored juices, they did not show any significant strange, negative sensory effects on the overall juice flavour. Only furaneol, which by its increasing in content, and its growing odour intensity during storage gradually contributed to a more pronounced caramel to honey taste of the juice. So, at the end of the 4-month storage of the juice, its flavour was a bit lacking in the typical, pleasant freshness of oranges.

Comparison of aroma profiles of samples from two production years indicated, that diverse climatic conditions, as well as the different seasons of oranges harvest in individual years could significantly affect organoleptic quality of fresh juice. Moreover, the inter-annual variability in such parameters of juice as the content of pulp and pH, could also affect the acid-catalysed reactions taking place in matrix during pasteurization and storage in a different way. This could lead to the observed different extent of content and odour intensity changes in the aroma profiles of orange juices from 2 years, more specifically, the processing and storage had lesser impact on the volatiles of orange juice that was produced in 2018, as in the following year.

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Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

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