Influence of protective inert gas atmospheres on the aroma stability of orange juice with pulp

JANA SÁDECKÁ[1], Mária Kopuncová[1,2], Emil Kolek[1], Jaroslav Blaško[2], Martin Polovka[1], Pavel Farkaš[3] and Ján Durec[4]
[1] *NAFC-Food Research Institute, Priemyselná 4, SK-824 75 Bratislava, Slovakia*
[2] *Faculty of Natural Sciences of the Comenius University, Institute of Chemistry, Ilkovičova 6, SK-842 15 Bratislava, Slovakia*
[3] *Axxence Slovakia, s.r.o., Mickiewiczova 9, SK-811 07 Bratislava, Slovakia*
[4] *McCarter, a.s., Bajkalská 25, SK-821 01 Bratislava, Slovakia*

Abstract

The efficacy of three divers processing atmospheres: nitrogen (N_2), carbon dioxide (CO_2), and conventional “air” (O_2) (as a control), for the protection of the volatile aroma compounds of commercially produced orange juice during its guaranteed four-month shelf life was investigated over two successive production years. Headspace-solid phase microextraction (HS-SPME), gas chromatography-mass spectrometry (GC-MS), and in parallel gas chromatography coupled to the flame ionization detector and with olfactometry (GC-FID/O) were used for the isolation, and subsequently for investigation of the volatiles with the emphasis on the key odour-active constituents of the aroma. Gained results showed that inert processing atmospheres can partly preserve the aroma profile of the orange juice. The best results were obtained with N_2 application. Concerning the outcomes of CO_2 application on orange juices, the results were comparable with N_2, but acceptability of juices treated by CO_2 have to be considered by consumers because of the sparkling character of final products evoking more sour taste. Nevertheless, none of two investigated inert gases was able to avoid all changes in the composition of volatiles during the storage time. However, from a sensory point of view, GC-FID/O analyses proved that these changes are not significant to that extent to lead to deterioration in the overall flavour of juices. On the contrary, certain negative sensory changes were observed for juices processed in conventional “air” (O_2) atmosphere as early as in second month of four-month shelf life, and they were getting worse gradually over the storage time. GC-FID/O revealed that the generation of some aldehydes, mainly hexanal, nonanal and perillaldehyde, as a consequence of oxidative changes could be responsible for this off-flavour phenomenon. In these juices obvious increase in bitter and waxy odour and taste was noticed, as well as the appearance of considerable astringent taste, a certain loss of freshness and fruity sweetness, and undesirable colour changes.

Introduction

Generally, the flavour of fresh hand-squeezed orange juice is considered to be the most attractive one, and it is used as a reference etalon against which all other types of orange juices are judged. Nevertheless, sensory perception evoked by commercial produced orange juice can be quite different because individual stages of industrial processing (freezing/thawing, depulping, deaeration, pasteurisation of raw juice) [1-9], influence of used packaging materials, as well as long-term storage in retail chain (impact of temperature, time, oxygen content, light exposure) result in some alterations in original fresh juice aroma [10-15]. It is obvious that a lot of effort has been devoted to the research of commercial orange juice up to now, so one potential way how to reduce degradation of fruit juices during storage can be their production under inert atmosphere.

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Experimental

Materials

Raw, unconcentrated orange juice imported from Costa Rica in frozen state was obtained, and afterwards technologically processed by McCarter a.s., Bratislava, Slovakia. After unfreezing, juice was enriched with pulp, mixed, pasteurised at up to 95 °C during 20 s and filled aseptically into the 200 ml polyethylene terephthalate (PET) bottles with oxygen scavengers. The first year, one series of samples from the same batch of raw juice was processed under N_2 atmosphere, and the second one by the traditional technology in conventional "air" (O_2) atmosphere. In the second year, one series of samples was produced under N_2, and the second one under CO_2 atmosphere. Bottled samples were stored in lab at 7±1 °C in the showcase refrigerator under conditions simulating the daylight exposure, i.e. typical conditions in a retail chain, within 4-month shelf life period. Analyses were performed in 24 h after delivery of samples to the lab, and then on a monthly basis.

Head-space solid phase microextraction (HS-SPME)

Each sample of orange juice (5.0 ml) was incubated statically in a 40 ml glass vial in a metallic block thermostat at 35 °C for 30 min, with a SPME fibre with 50/30 µm DVB/Carboxen/PDMS film (2 cm stable flex) placed in the headspace of sample. HS-SPME isolates were desorbed at 250 °C in GC injector during the entire GC runs.

Gas chromatography-mass spectrometry (GC-MS)

Obtained complex mixtures of the volatiles were analysed by GC-MS using the gas chromatograph Agilent 6890N coupled to the mass spectrometric detector 5973 inert equipped with fused silica capillary GC column Ultra 1 (50 m × 0.32 mm × 0.52 µm) operating with a temperature programme 35 °C (2 min), 4 $°C.min^{-1}$, 200 °C. The linear velocity of carrier gas helium was 33 $cm.s^{-1}$ (measured at 143 °C). Splitless injection mode was used at an injector temperature of 250 °C. Ionization voltage (EI) was set to 70 eV. Identification of compounds was performed by comparison of measured mass spectra with available mass spectral libraries Wiley and NIST MS. Relative proportions of individual volatiles as semi-quantitative parameters were calculated by the method of internal normalization and expressed as a percentage; the values were the averages of triplicates (data not shown).

Gas chromatography-olfactometry (GC-FID/O)

GC-FID/O was involved using the detection frequency concept of posterior evaluation of odour quality and odour intensity of individual odorants. A sniffing panel was formed from 5 sniffers who were chosen from 11 well-trained assessors in sensory analysis. Results of these analyses were expressed as the average values of estimated odour intensities in a scale from 0 to 3 with increments of 0.5, obtained from 5 independent measurements. Each sensory perception was based on at least 4 citations. The value ±0.5 was considered as measurement deviation. For the performance of these analyses, as well as for the determination of linear retention indices the gas chromatograph Agilent 7890A was coupled to FID and an olfactory detection port (ODP3, Gerstel). GC column Ultra 1 (50 m × 0.32 mm × 0.52 µm) operated with the temperature programme 35 °C (2 min), 4 $°C.min^{-1}$, 200 °C. Hydrogen was used as a carrier gas at the linear velocity of 44.6 $cm.s^{-1}$ (measured at 143 °C). Splitless injection mode was used at injector temperature of 250 °C. The linear retention indices (LRI^{U1}) were calculated according to the equation of Van den Dool and Kratz [16], using n-alkanes C_6–C_{14} as reference compounds. For GC-FID/O experiments the effluent of the column was splitted with a ratio of 1:1 to the FID and ODP, which operated at the temperature of 180 °C, interface temperature was 230 °C, the flow of added N_2 in ODP humidifier 12 $ml.min^{-1}$. The sniffing time of each judge did not exceed 30 min.

Results and discussion

GC-FID/O study of juices produced under N_2 atmosphere vs "air"(O_2) atmosphere

GC-FID/O technique was used in order to detect and identify volatiles which can be responsible for the sensory differences observed between juices processed in inert and conventional "air" atmosphere during storage, as well as to reveal potential off-flavour compounds causing negative changes in the aroma of juices. In general, 24 odour-active compounds were detected in the orange juice irrespective of used processing atmosphere (Fig. 1, Tab. 1), however, only 23 olfactory responses were recorded, due to the overlap between odours of octanal + β-myrcene. Odorants D-limonene, (Z)-β-ocimene, δ-3-carene, α-terpinolene[t], linalool, L-limonene[t] and decanal were principal in the volatile fraction of orange juice. They contributed with their high odour intensities (from 2 to 3) to the overall odour of orange juice to a decisive degree and thus, they were the most characteristic components of its odour. With regard to odorants such as (E)-2-hexenal, D-limonene, (Z)-β-ocimene, α-terpinolene[t], linalool, perillaldehyde and unknown compound No. 23, their odour intensities remained unchanged during the entire storage period in both processing atmospheres.

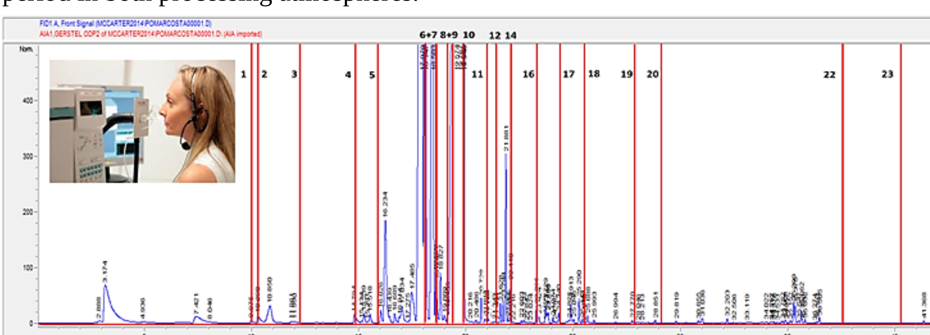

Figure 1: Gas chromatogram + olfactogram of orange juice volatiles (production without inert gas (O_2), 0. month of storage) obtained by HS-SPME coupled to GC-FID/O. The numbering verticals, marking olfactory responses corresponds to Tab. 1.

On the contrary, hexanal (green, grassy, leafy and bitterish odour), and also nonanal (soapy, waxy, tallow-like odour) were detected only in samples produced in conventional $(O)_2$ atmosphere (Tab.1). Perillaldehyde (smoked, cumin, spicy odour) was noticed in both atmospheres, but in conventional one showed higher odour intensity. Intensity of decanal (orange peel-like, waxy odour) dropped in N_2 atmosphere, whereas in conventional one was stable. Only undecanal (fatty, citrus, aldehydic, waxy odour) showed increasing trend in both atmospheres. Concerning the observed changes in odour intensities of some aldehydes, they can explain deterioration of the organoleptic properties of juice processed in conventional atmosphere that occurred during the second, but especially the third month of storage. Mainly, it was increased bitter and astringent taste of juice, it was registered a certain loss of freshness and fruity sweetness, accompanied by undesirable colour changes. In contrast, juice processed in N_2 atmosphere had standard organoleptic quality comparable to the fresh product during the entire storage period.

Table 1: Principal odorants of industrially processed orange juice with application of N_2 or without inert gas (0_2), revealed by the method HS-SPME coupled to GC-FID/Olfactometry

No.	LRI U1	Compound	Odour intensity during storage										Odour description	References
			0 month		1 month		2 months		3 months		4 months			
			O_2	N_2	O_2	N_2	O_2	N_2	O_2	N_2	O_2	N_2		
1	772.4	hexanal	0.5	–	0.5	–	0.5	–	0.5	–	0.5	–	grassy, leafy, green, slight fruity	LRI, MS, ST, OD, LIT
2	782.9	ethyl butanoate	1	1	1	1	1.5	1.5	1.5	1.5	1.5	2	fruity, apple-like, sweet	LRI, MS, ST, OD, LIT
3	822.8	(E)-2-hexenal	1	1	1	1	1	1	1	1	1	1	green, leafy, apple pip-like, slightly bitter	LRI, MS, ST, OD, LIT
4	926.6	α-pinene	1	1	–	–	0.5	0.5	–	–	0.5	–	sharp, pine, terpenic	LRI, MS, ST, OD, LIT
5	979.1 981.8	octanal β-myrcene	1	–	1	–	1	0.5	1	0.5	1	1	herbaceous, bitterish, terpenic, hop oil-like	LRI, MS, ST, OD, LIT
6	1001.3	δ-3-carene	2	2	2	2	1	2	1	2	1	2	turpentine-like, sweet citrus, sharp	LRI, MS, ST, OD, LIT
7	1005.9	α-terpinene	0.5	–	0.5	–	0.5	0.5	0.5	0.5	0.5	0.5	balsamic, herbaceous, marjoram-like	LRI, MS, ST, OD, LIT
8	1008.6	p-cymene	1	–	1	–	1	1	1	1	1	1	citrus-peel, fresh, weak fuel-like	LRI, MS, ST, OD, LIT
9	1018.1	D-limonene	2	2	2	2	2	2	2	2	2	3	citrus, terpenic, intensive citrus-peel odour	LRI, MS, ST, OD, LIT
10	1018.5	(Z)-β-ocimene	2	2	2	2	2	2	2	2	2	3	lime, green, sweet, lemon, orange	LRI, MS, ST, OD, LIT
11	1054.3	1-octanol	1	1	1	1	1	1	1	1.5	1	1.5	herbaceous, earthy, waxy	LRI, MS, ST, OD, LIT
12	–	α-terpinolene[t]	2	2	2	2	2	2	2	2	2	2	mushroom-like, plastic	MS, OD, LIT
13	1081.4	nonanal	–	–	1	–	2	–	2	–	2	–	soapy-fruity, waxy, tallowy	LRI, MS, ST, OD, LIT
14	1083	linalool	2	2	2	2	2	2	2	2	2	2	refreshing, floral, fragrant	LRI, MS, ST, OD, LIT
15	1102.2	ethyl 3-hydroxyhexanoate	–	–	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	smoky, leather, tobacco	LRI, MS, ST, OD, LIT
16	–	L-limonene[t]	2	2	2	2	2	2	1	1	1	1	intensive fresh floral, rose, sweet orange	OD, LIT
17	1157.1	terpinen-4-ol	1	–	1	1.5	2	1.5	1	1	1	1	earthy, woody, musty, waxy	LRI, MS, ST, OD, LIT
18	1180.3	decanal	2	2	2	2	2	1	2	1	2	1	orange peel-like, waxy	LRI, MS, ST, OD, LIT
19	–	perillaldehyde[t]	1	0.5	1	0.5	1	0.5	1	0.5	1	0.5	fresh, herbal, cumin, spicy	MS, OD, LIT
20	1285.3	undecanal	0.5	0.5	0.5	0.5	1	1	1	1	1.5	1.5	fatty with orange and rose undertone, waxy	LRI, MS, ST, OD, LIT
21	1360	geranyl acetate	–	0.5	–	0.5	–	0.5	–	0.5	–	0.5	fresh, green, lavender	LRI, MS, ST, OD, LIT
22	–	δ-cadinene[t]	1	–	1	–	1	–	1	–	1	–	thyme, slightly sweet, herbal, woody	MS, OD, LIT
23	–	unknown[o]	1	1	1	1	1	1	1	1	1	1	pleasant, floral, slight fruity, conditioner-like	–

Compounds identified on the basis of following criteria: LRI ^{U1}–linear retention index measured on GC column Ultra 1; MS(EI)-mass spectrum; ST-comparison with the reference compound; OD-odour quality; LIT-literature reference. [t]- tentative identification (only on the basis of mass spectra); [o]- compound detected only by GC-O

One of the principal findings of the study is that production of juices under inert atmosphere N_2 or CO_2 can protect their standard organoleptic quality from undesirable changes caused by oxidative load or acid-catalysed reactions during the guaranteed storage period.

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